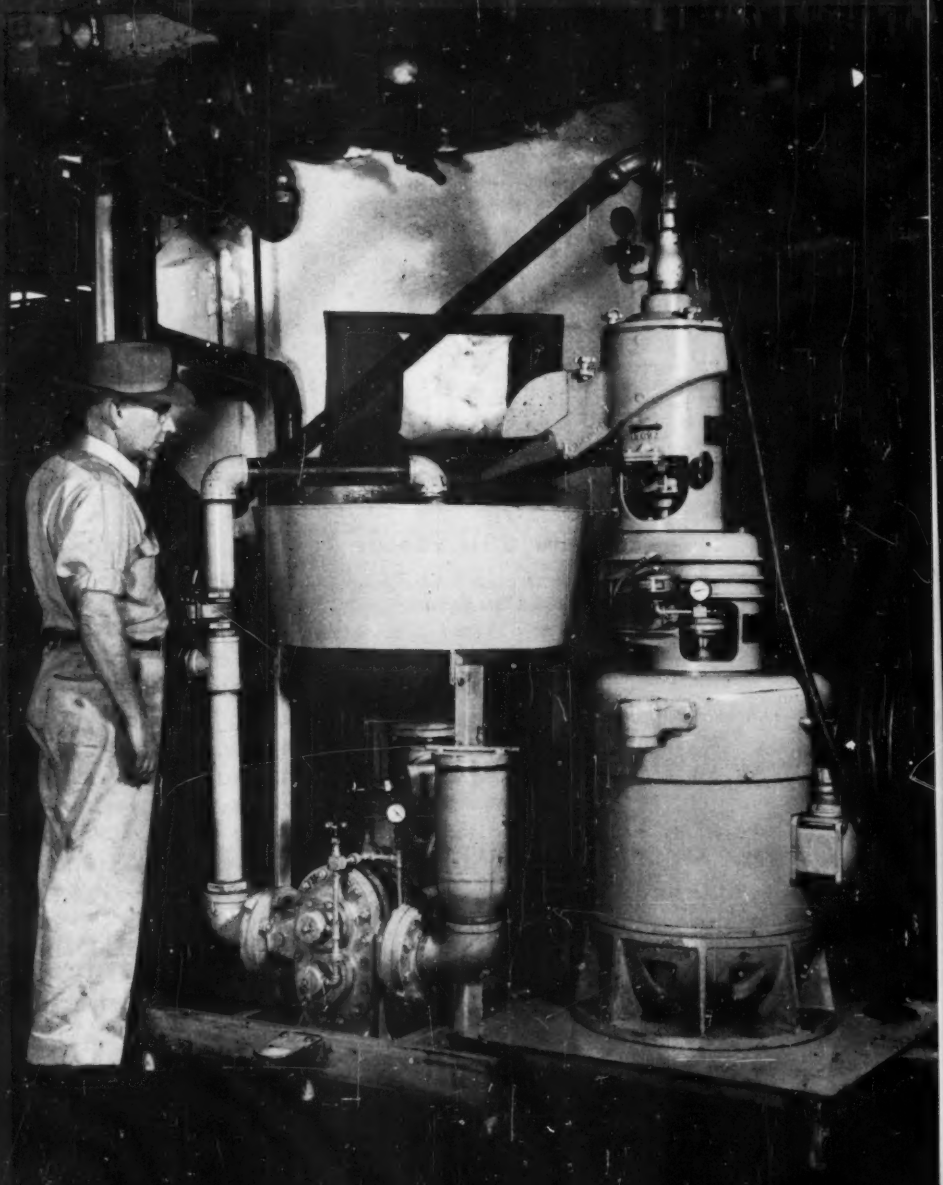
The Spokesman

MARCH



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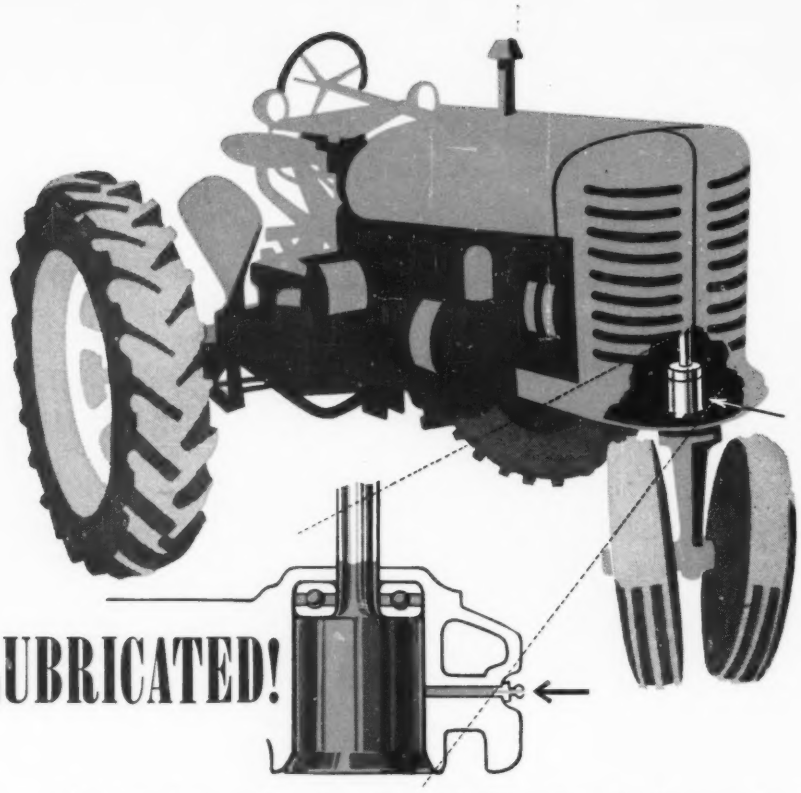
NATIONAL LUBRICATING GREASE INST. NLGI



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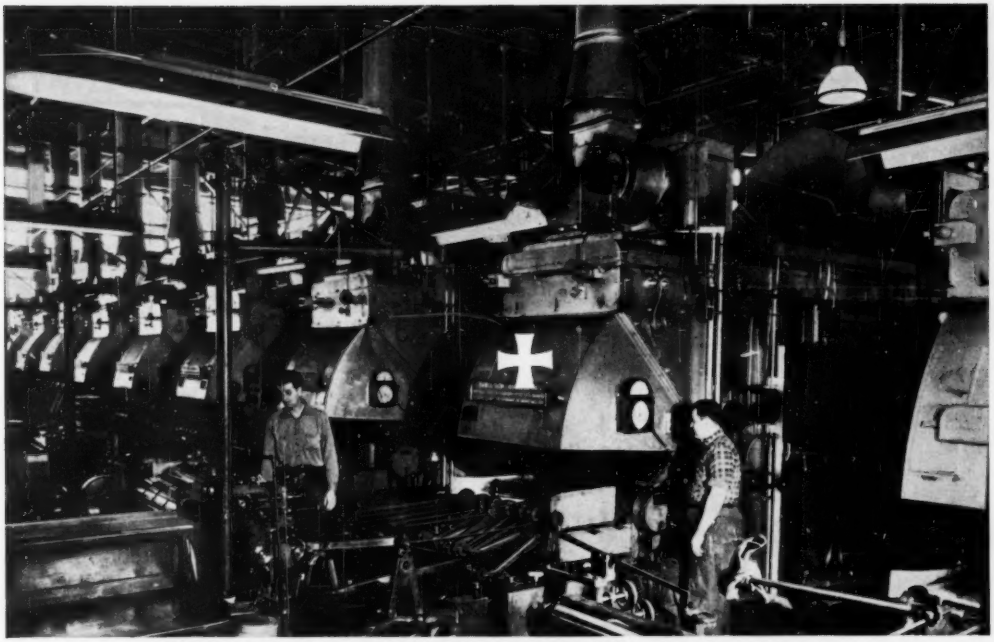

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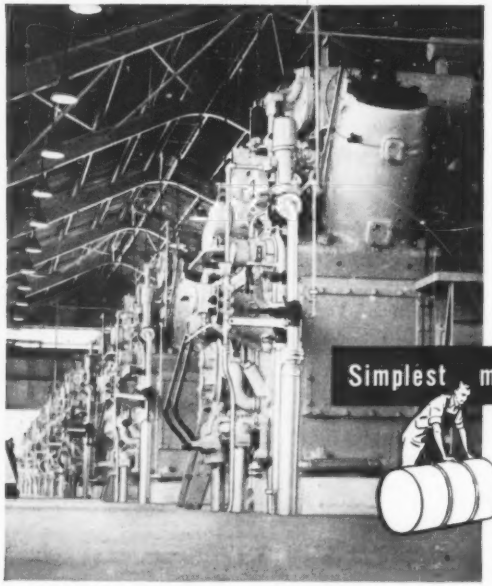

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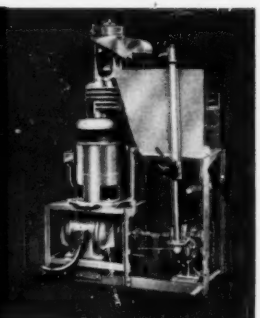

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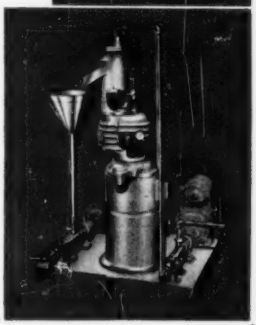

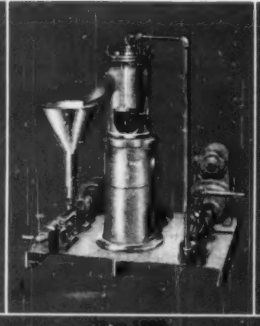

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President's page

by W. Wayne Albright, President, N L G I

NLGI AND GREASE RESEARCH

With the ultimate view toward improving lubricating grease products and their role in lubrication, the NLGI participates in research in a number of ways. One of our objectives, stated by J. R. Battenfeld at the first NLGI meeting in 1933, is "Collaboration with other scientific organizations."

How Objective Is Attained

How well this objective is attained may best be determined by consulting the pages of "The Institute Spokesman." Reports on lubricating grease research are published from time to time from a number of different sources. NLGI members having research facilities, other professional organizations (AFBMA, ASTM, ASME, NSA, AISE, and AAR for example) conducting research on lubricating greases or subjects associated with them, large grease users, Government research projects on greases and allied subjects, all make available to our members information on lubricating grease developments.

Another source of basic information on lubricating greases is our NLGI Fellowship program. Dr. Marjorie Vold, NLGI Fellow, presented her paper on X-ray diffraction studies of oriented soap structure in grease-like systems at our 1952 annual meeting. The NLGI Fellowship was established specifically for basic research in colloidal science.

All of this research information on lubricating greases and allied subjects is offered to NLGI members as one of our principal services. The result is improved grease products and better grease application techniques.

You Can Spread the Word

The general public is not aware of the amount and quality of research currently applied to greases and the importance of this research in development of products to do a better job of grease lubrication. There are several ways in which NLGI members can more widely publicize the research role of the grease maker in producing better lubricating greases. Institutional and product advertising, publicity releases, technical stories in trade magazines, technical company publications all can help to associate grease research with better grease products in the minds of the public. Copies of "The Institute Spokesman" can be used advantageously to help spread the word. Publicity of this sort is beneficial to both the maker and the consumer of lubricating greases.

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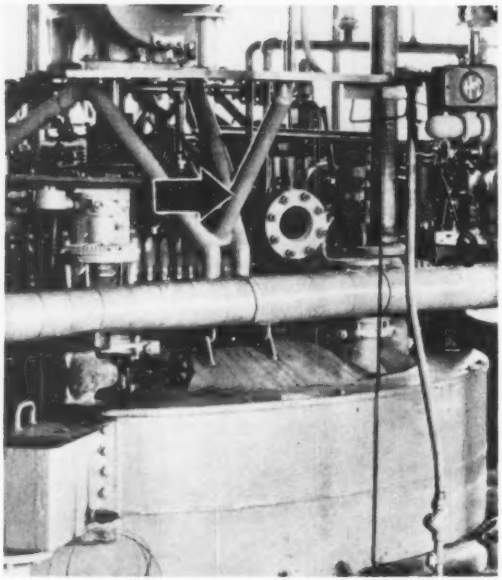
ABOUT THE COVER

The cover shows a comparatively recent installation of portable Morehouse Model B-2005 milling and deaerating equipment at the Vernon, California, grease plant of the General Petroleum Corp., a Socony-Vacuum company.

The unit has been in service for approximately nine months. It is used for track roller lubricant and other types of greases, resulting in increased yield, better appearance, and greater uniformity in the products handled.

The unit has a capacity of 25,000 lbs. per hour, is completely explosion proof, and can be disconnected and moved quickly and easily under the discharge pipes of one of several 18,000 lb. capacity mixers. This plant has used Morehouse equipment continuously since May, 1950.



GLYCERINE is used as the lubricant in the counter balance system (right of arrow) of a vinyl chloride reactor, where the lubricant's resistance to solvent-vapor attack is of utmost importance.

Varied Role Of Glycerine In Modern Lubrication

— a literary survey

by Milton A. Lesser
G. M. Basford Company
New York City

GLYCERINE has had many well-established uses in the field of lubrication for several decades. With the development of more specialized lubricants to meet the exacting requirements of modern equipment, glycerine has continued to be a highly versatile raw material for newer lubricant formulations. Today, applications for glycerine-containing products range from the lubrication of fine laboratory instruments to that of powerful Diesel engines.

Valuable by itself for many lubricant applications, glycerine is also of importance as a component of, or additive to, many greases and related compounds. The reasons for such wide usage are readily apparent when one considers the characteristics inherent in glycerine.

Primarily, glycerine's usefulness as a lubricant derives from the fact that it has the requisite body and oiliness required to combat friction. This is indicated in the studies made by Fogg and Hunwicks[1] in which the "boundary lubricating" properties or oiliness of various substances were obtained by means of a modified Deeley machine. The average values for glycerine, reported as coefficients of static friction, were 0.20 at 20° C. to 0.25 at 100° C. These may be compared with the values for light machine oil, which were 0.16 at 20° to 0.19 at 100°; or with those of solvent refined oil, which were 0.15 at 20° to 0.20 at 100°.

The wide range between the freezing point and boiling point of glycerine makes for its effectiveness as a lubricant under a wide range of conditions, especially at low temperatures. It is without fire or explosive hazards, and it is also a wholesome and non-toxic substance. The latter property helps to account for its frequent use as a lubricant for food processing equipment as well as for diagnostic and surgical instruments. The fact that glycerine is kind to rubber is also a factor in certain of its specialized applications.

Of major importance is the ability of glycerine to stand up under the action of hydrocarbon and other organic solvents and vapors that remove ordinary lubricants. This characteristic is recognized early in the career of the lubrication engineer or oil chemist when, as a student in the laboratory, he uses glycerinized lubricants for glass stopcocks, ground glass joints, vacuum pumps, stirrers and other apparatus.

Glycerine has found similar application in industrial lubricants. It is used, for instance, as a lubricant in special pumps for handling propane and butane. In these pumps, lubrication and sealing at the gland are provided by a reservoir containing glycerine that entirely surrounds the stuffing box and prevents gas from reaching it.

Glycerine-containing compounds are likewise employed for the stuffing boxes of high duty agitation equipment on pressure reactors and the like. The following basic lubricant has been recommended for use where solvent vapors such as carbon tetrachloride, vinyl chloride, petroleum ether, or the like, are predominant[2]:

Potassium soap (surgical soap-titer 20)	16 parts
Glycerine	4 parts
Water	1 part

This basic lubricant can be modified to meet various requirements. To make a paste for use when installing packing, the basic lubricant is thickened with sufficient powdered mica and asbestos powder. For use in semiautomatic and fully automatic lubricators, water is added to the basic lubricant until it is sufficiently fluid.

Of a similar nature are patented lubricants consisting basically of mixtures of soaps with glycerine[3]. Such compositions are suitable for the lubrication of taps and other moving parts in pipe systems carrying a hydrocarbon gas that liquefies under pressure. Glycerine is used extensively in the production of grease-like lubricants which are resistant

to hydrocarbons and suitable for use at low temperatures (e.g. below —100° F.). Such compositions are made by mixing glycerine with zinc stearate and a small proportion of a sulfonate type of wetting agent[4]. Somewhat different is a grease-like composition that is insoluble in hydrocarbon oils. Such a lubricant may be prepared by mixing glycerine with enough acetylene black to yield a product of the required consistency[5].

In addition to their use with solvent vapors, glycerine-based lubricants are also employed on the moving parts of containers or dispensers of explosive or corrosive gases. It has been stressed, for example, that glycerine should be used for all lubrication of the mechanism of gas (e.g. acetylene and oxygen) welding equipment[6]. A lubricant suitable for use on stopcocks in the presence of chlorine gas may be prepared by heating 60 parts of glycerine and 30 parts of sucrose to 120° C. until dissolved. This mixture is then ground with 10 parts of graphite or talc in a heated mortar. The resulting thick, pasty lubricant will not extrude upon frequent rotations of the stopcock and will resist the gas for long periods, it is said[7].

As already mentioned, the familiar antifreeze properties of glycerine are brought to the fore when low temperature lubrication is needed. For example, in hardening rooms where cans of ice cream are conveyed at low temperatures, glycerine is used as the lubricant for the conveyor. The fact that glycerine is a safe, wholesome substance is also an important consideration in its selection for such uses. Because of these characteristics, glycerine is frequently used to lubricate the moving parts (other than the motor) of mechanical food processing equipment, such as mincers, juicers and mixers.

When glycerine and glycerine-containing formulations are used with rubber goods, they serve, not only as lubricants, but also as extenders for the life of the rubber items. This is in marked contrast to the action of mineral oil and similar lubricants which cause brittleness and deterioration of the rubber in a comparatively short time. Glycerine rates a high position as a lubricant for rubber parts and surfaces, and is employed as a lubricant in extruding processes, as a mandrel and core lubricant and as a mold lubricant. An extension of the latter application is seen in the use of pressure loads of small gears of the worm and hypoid type[8]. metals are cast in rubber molds. Glycerine is said to be particularly suitable and to yield superior results when used as a lubricant in the rubber-forming of light metals.

Dispersions of colloidal graphite in glycerine are also extensively used as lubricants for rubber surfaces. Similar dispersions are valuable as lubricants for oxygen compressors, stopcocks, and as special purpose lubricants. Glycerine-graphite preparations make valuable lubricants for low temperature work, as well.

The newer uses for glycerine include a recent method for producing lubricants designed to stand up under the high pressure loads of small gears of the worm and hypoid type[8]. The action of these noncorrosive, odorless, extreme-pressure lubricants depends on the use of certain finely subdivided crystalline solids in a liquid vehicle, such as glycerine. In addition to acting as a carrier for the solid and as a coolant and heat-distributing agent, the glycerine serves as a polar liquid to cause adhesion of the solid to the metal surfaces being lubricated. Of fairly simple composition, a typical lubricant of this kind consists of 10 per cent molybdenum disulfide in a 90 per cent solution of glycerine in water. In other examples, a small proportion of a soap is included with the solid and the glycerine. Unlike other preparations of this type, these new lubricants are not used as additives to hydrocarbon oils, but rather as replacements for them in gear lubrication.

Glycerine is an important component of many modern greases made with hydrocarbon oils and soaps. This is shown by the fact that there are established procedures, such as the A.S.T.M. method of analysis, for determining glycerine in lubricating greases. The glycerine, which may be present as a by-product of saponification during the manufacture of a grease or as an additive, serves a number

FINISHED ICE CREAM is stored in giant lockers where the temperature is held at 20 degrees below zero. Glycerine is the recommended lubricant for the conveying equipment shown in the foreground.

ALTHOUGH GLYCERINE is now being made synthetically from the petroleum hydrocarbon, propylene, most commercial glycerine is still produced as a by-product in the manufacture of soap. Shown above is an 8000-gallon soap kettle where melted tallow, animal greases, and fatty vegetable oils are reacted with alkali. This reaction splits each molecule of fat into a molecule of glycerine and three molecules of sodium soap. Some soap-making kettles are as much as 33 feet deep.

of useful purposes in these lubricants. Glycerine aids in stabilizing the structure of the grease, reduces the change in consistency, increases the lubricating power, and prevents increase in the coefficient of static friction on heating. It has also been found that the presence of glycerine lowers the coefficient of friction at all temperatures below 90° C. and causes a greasy film to be deposited at high temperatures.

Additional research has further clarified the role of glycerine in greases. Studies on sodium soap-mineral oil systems have shown that the addition of glycerine increases the surface activity of the soaps at a given concentration, but the temperature at which the surface activity is attained remains unaffected. The presence of glycerine in a grease having a soda base also enables a nonpolar oil to wet the soap. In regard to the fiber structure of greases, glycerine is essential for the production of long fibers in ordinary practice, because the oil can wet the soap in its presence. Polar compounds, such as glycerine, promote fiber growth by facilitating the recrystallization from the gel form.

The technical and patent literature offers an increasing number of examples of the use of glycerine in lubricating greases. Thus, according to one report[9], the high temperature usefulness of soda-base greases, as well as their texture, is improved by the addition of a plasticizer, such as glycerine. A basic formula for such greases calls for the use of:

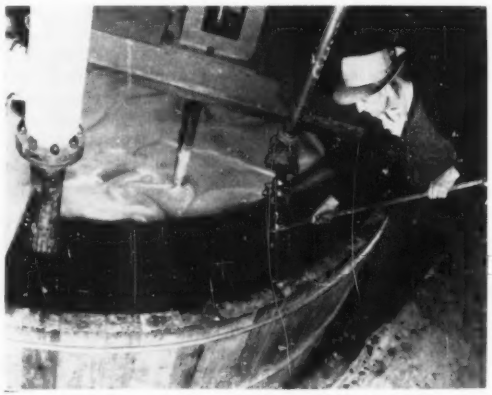
Tallow soap	40 — 50 per cent
Oil	40 — 50 per cent
Glycerine	2 per cent

Glycerine's utility with greases containing other metallic soaps has also been shown. For instance, cohesive greases, suitable for lubricating chassis bearings and the like, may consist of oil, aluminum stearate, polymerized isobutylene as a thickener, and up to two per cent of glycerine. In another case, the addition of glycerine to greases containing aluminum soap compositions is said to reduce the difference between the unworked and worked penetration values and to improve the resistance of the greases to working[10].

In another new development, up to three per cent of glycerine is utilized as a dispersant in the formulation of extreme pressure lime-base greases containing a lead soap[11]. Also indicative is the use of glycerine, together with a lithium or other metallic soap and a silicone polymer, for the production of lubricants that are particularly useful for specialty equipment employing shielded or sealed bearings and operating under wide temperature ranges. A typical example is formulated as follows[12]:

	per cent
Lithium soap	12.8
Glycerine	0.9
Mineral Oil	13.4
Dimethyl silicone polymer	72.9

Other more specialized lubricating grease formulations make use of the various improved properties provided by glycerine. For example, glycerine is listed first among the



SOAP-MAKING in process. When the saponification, or fat-splitting, reaction is complete, rock salt is added to make the soap separate from the liquid. Soap forms the upper layer; glycerine in solution with salt and water is drawn off from the bottom of the tank.

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polyhydric alcohols suitable for imparting water emulsifiability to stainless greases that are used in pipe-bending and wire-drawing operations[13]. In another instance[14], glycerine is used in the preparation of soluble oil bases that impart rust-preventing and corrosion-inhibiting characteristics to metal-working lubricants.

Before concluding, mention should be made of the potentialities of glycerine-containing compositions as antifoaming agents for lubricants. In investigations sponsored by the National Advisory Committee for Aeronautics[15], it was shown that mixtures of glycerine and a surface-active agent (Aerosol OT) were completely effective in controlling foaming in aircraft engine oils. Similar combinations of glycerine with wetting agents are employed as additives to lubricants to combat the tendency of oils and oil compositions to foam in the presence of gas, air, steam, oil vapor, products of combustion, or the like[16]. Dispersions of these antifoaming agents are said to abate or suppress the normal foaming tendencies for long periods without deleterious effect on the other properties of the lubricants. Moreover, only small amounts are needed. For exmaple, the addition of 1.0 per cent of the following mixture to S.A.E. 50 oil reduced foaming to zero in five minutes under the test conditions:

Dioctyl sodium sulfosuccinate (Aerosol OT)	5 parts
Glycerine	95 parts

Such applications, as well as others previously cited, show how glycerine's properties may be adapted to solve both simple and difficult problems of the lubricating industry. Lubricating engineers and oil technologists are becoming increasingly aware of the high degree of usefulness inherent in glycerine, and they are making increased use of this fluid to improve old products and to create new ones to meet ever-changing requirements.

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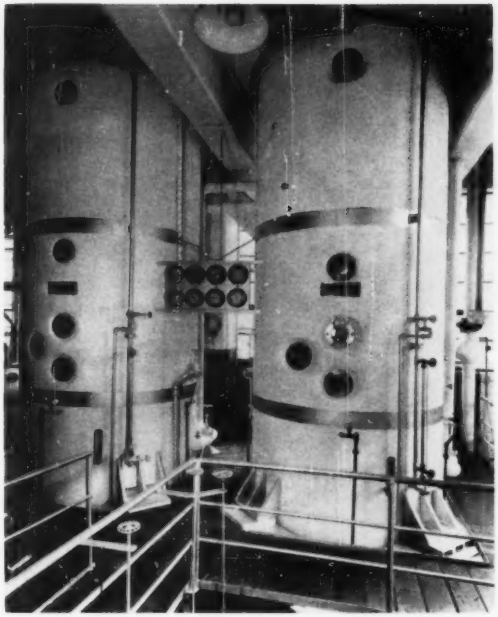


THE MIXTURE OF GLYCERINE, WATER, SOAP, AND DILUTE ALKALI from the soap kettles is first treated with aluminum sulfate and filtered to remove dissolved soap. The solution is then concentrated under reduced pressure in tanks such as the above Wurster & Sanger double effect evaporator. As water is removed, salt separates out in a salt box at the bottom of the tank. The final product is "soap lye crude glycerine" which contains about 80 per cent glycerine.

5. Morway, A. J. & Zimmer, J. C.; U. S. Pat. 2,467,146, 1949.

6. Anon; Ohio Indust. Monitor, October 1949, p. 158.

7. Muirhead, G. S.; Chemistry & Industry 1947, p. 628.

8. White, J. R., Clark, O. H. & Woods, W. W.; U. S. Pat. 2,609,342, 1952.

9. Anon; Iron Age, 152:73, October 28, 1943.

10. Ashley, K. D. & Mason, S.; Pat. 2,555,104, 1951.

11. Schott, J. E. & Armstrong, E. L.; U. S. Pat. 2,583,394, 1952.

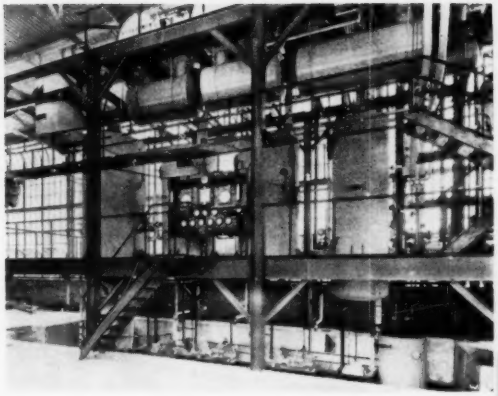
12. Ashburn, H. V., Barnett, R. S. & Puryear, O. P.; U. S. Pat. 2,508,741, 1950.

13. Showalter, J. C.; U. S. Pat. 2,599,353, 1952.

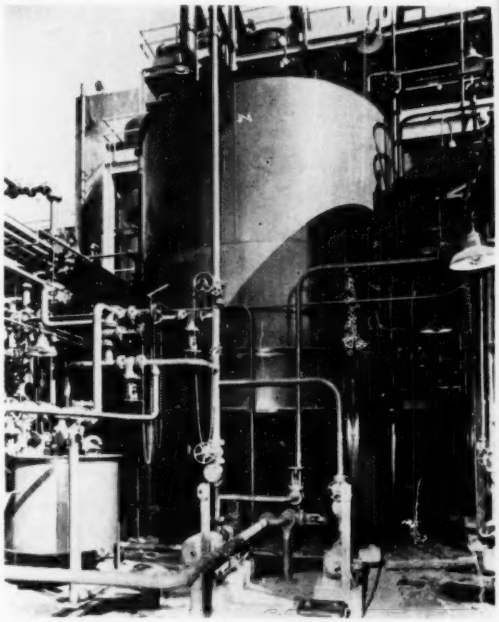
14. Schiermeier, K. F.; U. S. Pat. 2,516,838, 1950.

15. McBain, J. W., Ross, S., et al; NACA Technical Notes Nos. 1841 & 1842, Washington, National Advisory Committee for Aeronautics, April, 1949.

16. Trautman, C. E.; U. S. Pat. 2,603,599, 1952.



A CONTINUOUS GLYCERINE DISTILLATION PLANT where crude glycerine from the evaporators is purified by distillation under reduced pressure is shown above.



AN ION-EXCHANGE UNIT — a development which takes the place of the evaporation step is pictured. Here, dissolved salts, acids, alkalis, color bodies, and odor are all removed from crude glycerine solutions by the use of ion-exchange resins. No heat is required. After this treatment, the purified solution is distilled to remove the water.

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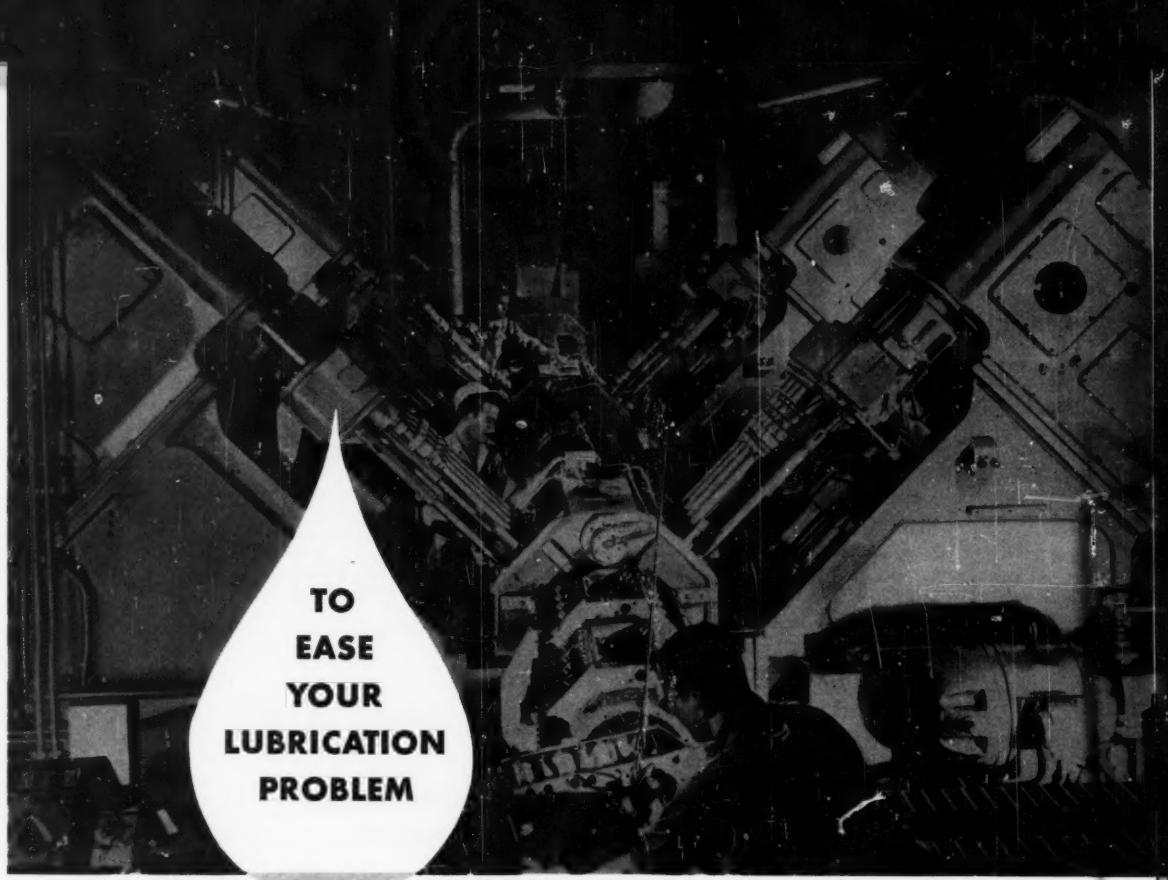

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Symposium On Utilization Of Fatty Acids In the Manufacture Of Lubricating Greases

Papers Presented at the NLGI Technical Committee Meeting, October 29, 1952

Introduction

T. E. DeVilliers, Committee Chairman,
Cities Service Oil Company

IT is recognized by lubricating grease technologists that the type of soap incorporated in a lubricating grease composition will affect its properties. This is true not only of the type of base used in making the soap, but of the composition of the fat or fatty acids as well. The type of mineral oil used also will have a marked effect on grease properties, as will the manufacturing process. Certain grease properties may be varied extensively by the addition of selected materials in relatively small — although sometimes major — proportions. These latter materials are commonly classified under the rather loose term "additives".

A better knowledge of the variation in grease properties caused by variation in grease-making raw materials would benefit both the grease manufacturers and the suppliers of raw materials. Recognizing this, the Technical Committee held a symposium on the effect of fatty acids composition on grease properties at the 1952 annual meeting of N.L.G.I.

A symposium affords an opportunity for a number of persons with a common interest to gather for discussion of a selected subject. The authors are not limited to mere reporting of factual data but are free to offer opinions and ideas as they choose. One of the prime purposes is to stimulate constructive thinking on the subject under consideration. The symposium is an entity composed of various parts and the reader should not lift a part out of context and consider it authoritative. The authors have made many general statements and have not intended to present recommended processes and formulations.

Sources, Manufacture And Composition of Fatty Acids

J. M. Kiefer
Armour Chemical Division

Strictly speaking, the term "Fatty Acid" applies only to acetic acid and its homologs. However, usage, in this country at least, has given it a somewhat different connotation. As now generally used, the term "Fatty Acid" is applied to any of the aliphatic mono-carboxylic acids obtained by hydrolysis of animal or vegetable fats, oils or waxes. This usage departs from the strict definition in two ways. It goes beyond the latter in that it thus includes many related unsaturated acids, hydroxy-acids, keto-acids, and even some containing alicyclic substituents. On the other hand it restricts the formal definition in that it excludes the myriad of known and possible branched chain homologs of acetic acid. The acids derived from fats, oils and waxes are characterized, almost without exception, by normal — or unbranched hydrocarbon chains. It is in the sense of the presently accepted usage, that I use the term "Fatty Acids" in the present discussion.

Our having thus practically defined fatty acids as the acids obtained by hydrolysis of animal or vegetable fats, oil and waxes it should, of course, come as no surprise that these are actually the primary source of commercial fatty acids. As a matter of fact, this is the form in which many lubricating grease makers still buy their fatty acids, performing the hydrolysis themselves in the course of making a

grease. It is not the purpose of this discussion to argue the relative merits of glycerides versus fatty acids for grease making. That debate still goes on among your own members, who are much more qualified than we are to arrive at the answer. However, I will point out later some of the advantages in processing the glycerides to fatty acids before introducing them into the grease kettle. Either way, your grease making fatty acids start out as glyceride oils. There are literally thousands of different naturally occurring fats and oils. Perhaps 600 to 700 of them have been studied in some detail. But all of the commercially available fatty acids are derived from less than 50 different glyceride oils, and 10 of these would account for a major percentage of the total fatty acid production. These glycerides may be divided into groups having common characteristics. It should be noted here that the distinguishing characteristics of the glyceride oils are the direct result of their fatty acid compositions; the particular acids contained and the relative proportions of the different acids. The principal acids involved are as follows:

Saturated Acids:

Caprylic Acid	—	8	carbon chain
Capric Acid	—	10	" "
Lauric Acid	—	12	" "
Myristic Acid	—	14	" "
Palmitic Acid	—	16	" "
Stearic Acid	—	18	" "
Arachidic Acid	—	20	" "
Behenic Acid	—	22	" "

Unsaturated Acids:

Palmitoleic Acid	— 16	carbon chain;	1	ethylenic double bond
Oleic Acid	— 18	" "	1	" " "
Linoleic Acid	— 18	" "	2	" " "
Linolenic Acid	— 18	" "	3	" " "
Eleaostearic Acid	— 18	" "	3	" (Conjugated) " "
Erucic Acid	— 22	" "	1	" "
Various highly unsaturated Acids	— 20 - 26	carbon chain;	4 - 6	ethylenic double bond

Substituted Acids:

Rincinoleic Acid	—	12-hydroxy oleic acid
Licanic Acid	—	4-keto eleaostearic acid

The glyceride oils which serve as raw materials for commercial fatty acids may be grouped as follows: Lauric acid oils, animal fats, oleic-linoleic acid oils, linolenic acid oi's, conjugated acid oils, marine oils, and hydroxy acid oils.

Of the lauric acid oils, coconut oil is the chief fatty acid raw material. This group is characterized by a high content of the lower molecural weight saturated fatty acids, particularly lauric acid. These acids are desirable in many applications because of their lower melting points in relation to higher saturated acids such as stearic and palmitic, and particularly because of the greater water solubility of their soaps, even compared with those of the higher unsaturated acids.

The animal fats, sold industrially as tallows or greases, contain about equal amounts of saturated and unsaturated fatty acids. The saturated acids are primarily palmitic and stearic in a ratio of 60 to 40; the unsaturated acids consist chiefly of oleic acid, with a small amount of linoleic. The principal difference between tallow and grease is the somewhat larger content of linoleic acid in the latter. Palm oil is somewhat similar to the animal fats in composition, with the important exception that the saturated acid content is mostly palmitic acid. It is, of course, of vegetable rather than of animal origin. In these acid mixtures we have a combination of saturated and unsaturated acids which is highly desirable in soap and grease making.

Oleic-linoleic acid oils include many of the non-drying vegetable oi!s such as cottonseed, corn, and so forth. These oils generally contain minor amounts of saturated acids and practically no linolenic acid, their principal acid components being oleic and linoleic. Thus they yield fatty acids containing a high percentage of unsaturated liquid components without the strong tendency toward oxidation, discoloration and rancidity associated with the drying oils.

The linolenic acid oils are also vegetable oils but contain varying amounts of linolenic acid. The latter acid, you will recall, has three ethylenic double bonds and is therefore more susceptible to oxidation, rancidity, and polymerization. Examples are soybean oil, with a small amount of linolenic acid, and linseed oil, which often contains over 50% of this acid. Generally, these acids are used for their drying characteristics, although soybean acids are used for nondrying applications also, since the effect of the small amount of linolenic acid is not predominant.

The conjugated acid oils such as tung or oiticica oils contain large amounts eleaostearic or licanic acids, which are very readily polymerized by oxidation or heat, and are used exclusively for these properties. As a matter of fact, these oils are not widely processed into commercial fatty acids because of their sensitivity to heat.

Marine animal oils vary rather widely in composition but are characterized by their content of highly unsaturated high molecular weight acids having 20 to 26 carbon chain lengths and four to six double bonds. They also contain considerable amounts of saturated acids and, as a result, have only fair to poor drying qualities, while their high unsaturation makes them sensitive to rancidification and discoloration by oxygen. Hence, these acids have little utility unless the saturated acids are removed or the unsaturation is reduced by hydrogenation.

Of the hydroxy acid oils, we have only one of commercial significance, castor oil. It contains better than 85% ricinoleic acid. This acid has desirable properties unique among the fatty acids, with respect to solubility and chemical reactivity.

We cannot complete our discussion of sources of commercial fatty acids without mentioning "tall oil". This raw material is not a glyceride oil, but a mixture of fatty acids (primarily oleic and linoleic) and rosin acids. The fatty

acids may have existed as esters in their natural form in pine wood, but they come to us already hydrolyzed as an oily by-product from the sulfite process of paper making. This mixture offers a source of oleic and linoleic acids, with relatively little saturated acid content, if the accompanying rosin acids are not objectionable.

Another note on raw materials: Many of the vegetable oils, such as cottonseed, corn and soybean oils, are used in tremendous quantities for manufacture of salad and cooking oils and shortenings. For all of these uses it is necessary that the free fatty acids in the oils be removed by alkali refining. In this process the free acids are neutralized with caustic soda and the soaps formed are separated along with a certain amount of the occluded glyceride oil. This mixture of soap and oil is known as soapstock or foots. Refining foots are used, rather than the oils themselves, as the raw materials for producing the corresponding fatty acids. This fortunate circumstance allows the fatty acid industry to supply double distilled cottonseed, corn and soybean fatty acids at prices which are usually below those of the corresponding crude oils.

The processing of the above materials to produce commercial fatty acids varies considerably with the raw material itself and with the nature and quality of the acids to be made. However, there are several unit processes which are common to fatty acid operations. To obtain fatty acids from glycerides, hydrolysis of the esters is required. This process, often referred to as "splitting", is done in several ways. For many years, glycerides were saponified with caustic soda in a soap kettle and the soaps were then treated with acids to liberate the free fatty acids which are insoluble in water. This method may still be used in some cases but the more general procedure is the Twitchell method of acid hydrolysis wherein the glycerides are boiled with dilute acid, a surface active agent being used to provide good contact between the aqueous solution and the water insoluble glycerides. More recently, high temperature splitting has come into practice. Oil and water are continuously pumped countercurrent through a tower at a high temperature (500° F.) and of course at high pressure to maintain the water in the liquid state. At this temperature the oil is sufficiently soluble for hydrolysis to take place without use of catalyst, although a catalyst may be used in the process if desired. The last method is excellent unless exceptionally heat sensitive stocks are being split, in which case the milder Twitchell method must be used.

Catalytic hydrogenation of the ethylenic double bonds in unsaturated fatty acids is widely used. The acids may be completely saturated or, by carefully controlling conditions, partial hydrogenation of polyene acids may be accomplished. Thus, linoleic acid may be reduced to oleic acid, or to stearic acid. Finely divided nickel is the catalyst most generally used. While fatty acids themselves may be hydrogenated, reduction of the glycerides before splitting is preferred where practical.

Distillation of fatty acids is practiced with several ends in view. In some cases, distillation is carried out merely to improve the color and purity of the acids. In others, the acid mixtures are fractionally distilled to remove one or more components or to produce individual fatty acids of fairly high purity. In either case, distillation is carried out under reduced pressure in order to reduce distillation temperatures. Even in the simple distillation some fractionation is accomplished, in that lower boiling unsaponifiables and color bodies are stripped out and non-volatile unsaponifiables, glycerides, polymers and colored materials are left in the distillation residues. As a consequence, the distilled fatty acid mixtures, such as double distilled animal, cottonseed, corn, or palm acids, offer a lighter color and more uniform fatty acid composition than do the corresponding crude oils. Fractional distillation is carried out in specially designed bubble-cap-plate towers. By this method, fatty acid mixtures are separated into fractions containing a very high concentration of acid of a single carbon chain length. Among acids of the same chain length, differences in the degree of unsaturation do not produce sufficient difference in boiling points to make separation by this means commercially practical.

Crystallization, on the other hand, offers a means of separating fatty acids according to melting point or solubility. Here the differences due to degree of saturation in the fatty acids are quite pronounced. As a matter of fact, this process was probably the first separation procedure employed in the fatty acid industry; namely, the separation of animal fatty acids into red oil and pressed stearic acid. In the process, the mixed acids are chilled to partially crystallize them and then pressed to separate the liquid and solid portions. More recently, crystallization from solvents is being used, resulting in much better separations and eliminating the cumbersome pressing operation. Another processing technique based on solubility is that of extraction with liquefied propane. This process serves to remove oxyacids and results in some separation by degree of unsaturation also.

The foregoing processes are the basic tools of the fatty acid industry. They provide means for improving the quality of fatty acids, reducing the extent of unsaturation, separating or concentrating individual fatty acids with respect to their carbon chain length or degree of unsaturation. Let us see how these are applied in the production of specific commercial fatty acids.

Coconut oil is split to fatty acids which are straight distilled to give a mixture corresponding to that in the original oil or partially fractionated to remove the 8 and 10 carbon chain acids, caprylic and capric. This removal is desirable in the soap field because the lower molecular weight soaps are slightly irritating to the skin. The coco acid mixture is also fractionally distilled to produce high purity caprylic, capric, lauric and myristic acids.

Animal fats are split directly and distilled to produce double distilled animal fatty acids. By crystallization this mixture is separated into red oil, which is primarily oleic acid, and pressed stearic acid, which is a mixture of palmitic and stearic acids, palmitic being in slight excess. In alternative procedures, the animal fats are partially or completely hydrogenated before splitting. Partial, selective hydrogenation converts most of the linoleic and linolenic acids to oleic acid. When the resulting acids are crystallized, an oleic acid fraction is obtained which has a much lower polyunsaturated acid content than normal red oils. Completely hydrogenated animal fats yield a mixture of roughly 70% stearic acid and

30% palmitic acid. This mixture may be used as such giving a higher stearic acid content than the pressed stearic acids. The mixture is also fractionally distilled to obtain relatively pure palmitic and stearic acids. With these fractions, mixtures of palmitic and stearic acid in any desired ratio can be supplied.

The vegetable oils, usually obtained in the form of refining foots, are acidulated and split to hydrolyze the occluded glycerides. The acids are then distilled to give mixtures corresponding to the original glyceride composition. Such mixtures are double distilled cottonseed, corn, soybean, palm, linseed, peanut, perilla, rapeseed or sunflower acids.

Fractional distillation of cottonseed acids removes most of the palmitic acid, which is the principal saturated component, leaving a mixture of oleic and linoleic acids in about equal proportions with a low content of saturated acids. Solvent crystallization of the latter material or of double distilled soybean fatty acids yields mixtures of linoleic and oleic acids practically free of saturated acids and with better than 50% of linoleic. This high linoleic content with little or no saturated acids is desirable for making alkyd resins of the drying type. The mixture from fractionated cottonseed acids is very low in linolenic acid, and consequently has better non-yellowing properties, whereas that from soybean fatty acids has an appreciable amount of linolenic acid which results in slightly faster drying with greater tendency toward yellowing.

Tall oil as recovered from paper processing is already hydrolyzed to acids, but it is very dark in color. Simple distillation gives a product of good color but it still has the high rosin acid content. Fractional distillation yields an essentially fatty acid fraction consisting of oleic and linoleic acids, the content of the latter being about 40%. This mixture also contains small amounts of linolenic acid and rosin acids. A fraction high in rosin acids is also obtained.

Marine oils are split and distilled to give acid mixtures corresponding to that in the original oils. These are used in core oils, linoleum, etc. because of their highly unsaturated acids. Since most of the unsaturation is present in the high molecular weight fatty acids, these can be separated from the lower molecular weight saturated acids by fractional distillation, to give hard drying type acids.

For other uses the high degree of unsaturation is reduced by catalytic hydrogenation and the acids then distilled. These hydrogenated fish oil fatty acids differ from previously mentioned saturated acid mixtures in that they contain considerable amounts of arachidic and behenic acids. Fractional distillation of such mixtures can produce these higher molecular weight saturated acids in fairly high concentration.

Castor oil is split to produce ricinoleic acid or is first hydrogenated and then split to yield 12-hydroxy stearic acid. Castor oil or ricinoleic acid are also dehydrated, the removal of the hydroxyl group resulting in the formation of linoleic acid and a conjugated isomer of linoleic acid which has excellent polymerization and film-forming properties.

In addition to the above processing methods, which are primarily concerned with separation or alteration of the naturally occurring acids, there are several procedures which result in more fundamental changes in chemical structure and yield acids which are not strictly fatty acids according to our definition. Castor oil is pyrolyzed to give undecylenic acid. Ricinoleic acid heated with caustic yields sebacic acid, a 10 carbon dibasic acid. Oxidation of ricinoleic acid with nitric acid produced suberic and azelaic acids, which are respectively 8- and 9-carbon dibasic acids. Oleic acid is oxidized similarly to yield azelaic acid and pelargonic acid. The latter is a 9-carbon monocarboxylic acid. Under milder oxidation conditions, oleic acid can be oxidized to dihydroxystearic acid. Linoleic acid, heated under controlled conditions, is dimerized to a 36-carbon dibasic acid. And, of course, the fatty acids are processed to form a large variety of chemical derivatives which are no longer fatty acids, such as esters, alcohols, ketones, amides, nitriles, amines or chlorides, which are beyond the scope of this paper; but which should also be of interest to grease makers.

All of the foregoing presentation serves to show what is being done by the fatty acid industry to supply acids designed to meet specific requirements. Many industrial fields employing fatty acids (the protective coatings field is a good example) are no longer content to take fatty acids as natural glycerides, the good with the bad. They have, by painstaking investigation, found out which acids contribute to the quality of their product and which are detrimental. With this information at hand, the fatty acid producers have been able to supply materials which more nearly meet their requirements. Various N L G I members now have such programs under way and there can be no doubt that the information so developed will be of considerable advantage to the grease makers and fatty acid producers alike.

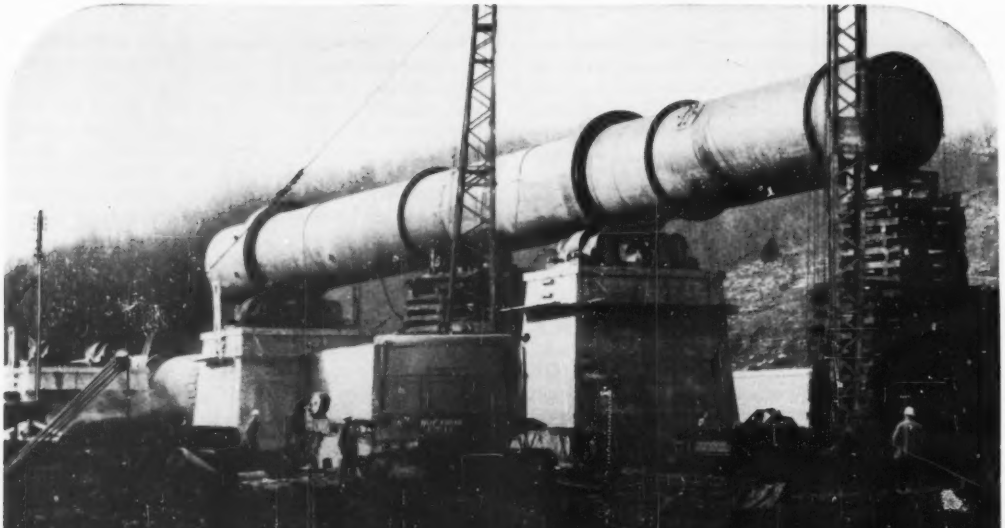
Analytical Methods In the Fatty Acid Industry

J. L. Trauth
Emery Industries, Inc.

Introduction

Fatty acids are becoming increasingly popular raw materials for the manufacture of lubricating greases. Providing a wider range of properties than the naturally occurring fats and oils, the many commercially available fatty acids can impart new utility and broader flexibility to lubricating grease manufacture.

The variety of commercially available fatty acids has increased rapidly in recent years, as the result of extensive research and development programs carried out within the fatty acid industry. New processes have brought forth many unique fatty acids which provide greasemaking characteristics difficult or impossible to attain using whole fats or oils. In addition to imparting novel characteristics, fatty acids provide the grease-maker with other important advantages, chief among which is uniformity. Fatty acids are manufactured and sold on the basis of rigid specifications, thereby offering valuable insurance against a faulty finished product. The grease-maker knows that the quality of his fatty raw material will be consistently uniform since composition, color and all other properties will be controlled within strict limits by the manufacturer. It is the purpose of this paper to discuss the analytical methods used by the fatty acid industry to maintain the high quality of its products.



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Product improvement has been the watchword of the fatty acid industry during the past decade and a number of new test procedures have been developed to measure such important characteristics as composition, resistance to atmospheric oxidation and color stability. These methods and many others, some already familiar to most of you, will be discussed as they are used during the course of fatty acid production.

Raw Materials—Tests and Purchase Specifications

Inedible fats and oils of animal, vegetable and marine origin are, of course, the starting materials for the manufacture of fatty acids. Inedible tallows and greases are used as raw materials for manufacturing animal fatty acids, stearic and oleic acids. These fats are by-products of the meat packing industry, and vary in quality from the very high grades such as fancy tallow and white grease to the very low grades such as No. 2 tallow and brown grease. Fancy tallow represents almost pure beef fat while white grease is essentially all pork fat. The low grade fats, however, are somewhat variable mixtures of beef and pork fat and are usually in an advanced state of hydrolysis. They are further characterized by high free fatty acid (F.F.A.) content, dark color and high unsaponifable content. Fats are accordingly graded according to MIU, color, free fatty acid and titer. MIU is the abbreviation used to designate the sum of the moisture, impurities and unsaponifiable. Purchase specifications include limits for all of these characteristics. In recent years there has been a trend in the fatty acid industry to add iodine value to its purchase specifications for animal fat to insure the exclusion of such highly unsaturated stocks as vegetable, horse and chicken fats which make it difficult, if not impossible, to maintain the required iodine value specifications on finished products.

The fatty acid industry also purchases considerable quantities of vegetable oils and vegetable soapstocks for the production of cottonseed, soya, corn, coconut and other fatty acids. Raw material specifications for vegetable oils include the same characteristics as those given for animal fats but special emphasis is placed on iodine value because of its importance in detecting possible contamination with other oils. Hydroxyl value or acetyl value is usually included in specifications for castor oil for the same reason.

At the present time the only requirements governing vegetable soapstock transactions are that the soapstock contain a specified minimum amount of Total Fatty Acid and usually a general statement to the effect that the soapstock must not be adulterated with any other oil or with soapstock made therefrom without consent of the purchaser. No provision is made for detecting possible contamination. Purchasers of vegetable soapstocks have found that with the introduction of improved refining techniques in the edible oil industry and the resultant concentration of impurities in the soapstock, it is necessary to have better specifications to insure reasonable freedom from contaminants and impurities which have a marked effect on the quality and yields of finished products. To this end the American Oil Chemists' Society (A.O.C.S.) was requested to devise a method for the evaluation of vegetable soapstock, and a soapstock analysis committee was accordingly created to carry out this assignment.

The fatty acid industry has found that vegetable soapstocks in general, and cottonseed soapstock in particular, having low total fatty acid content on a moisture-free basis, will generally give high residue yields with correspondingly low yields of very poor colored distillate. Such soapstocks contain a large amount of petroleum ether-insoluble but ethyl ether-soluble material, which is commonly referred to as "oxidized fatty acids". It has been observed that the oxidized fatty acid content is fairly indicative of both the yield and the quality of the distillate that may be expected from a soapstock. Generally speaking, the higher the oxidized fatty acid content, the lower the distillate yield and the poorer the distillate color and color stability. At present, the A.O.C.S. has no method for "oxidized fatty acids", but since there is an urgent need for such a test, the soapstock analysis committee is currently engaged in cooperative work on a method which looks very promising.

The fatty acid division of the Association of American Soap and Glycerine Producers has asked the National Cottonseed Products Association and the National Soybean Processors Association to revise their trading rules so as to include iodine value and unsaponifiable specifications for cottonseed and soya soapstocks. Limits for oxidized fatty acids will probably be included as soon as a suitable method is advanced by the A.O.C.S.

Marine oils are purchased on the basis of free fatty acid, moisture and impurities, but iodine value and color, if not actually specified, are always watched carefully by the buyer.

Quality Control of Fatty Acids During Production

The first step in the production of animal and vegetable fatty acids generally consists of acid washing the raw fat or oil in order to remove both dissolved and suspended impurities which might otherwise adversely affect subsequent processing operations and product quality. Since this step consists of boiling the fat with fairly concentrated sulfuric acid, some hydrolysis of the fat occurs, with subsequent glycerine loss. This operation is therefore controlled so as to get the required amount of purification with the least possible glycerine loss. This is accomplished by controlling the boiling time and acid strength. Glycerol content before and after boiling is checked by the periodate method to make certain that the glycerine loss does not exceed established limits. The glycerol content of the acid-washed stock is also used to determine the yield of glycerine recovery in the splitting and glycerine departments. Inventory control usually necessitates checking the moisture content of stock movements from storage to the acid boil department and from the acid boil department to the splitting department.

Splitting

Progress of the splitting or hydrolysis step is determined by getting periodic checks on the free fatty acid content of the stock as it comes from the column of a continuous pressure splitter, or as it completes the boiling cycle in a batch autoclave or Twitchell splitter. Per cent hydrolysis is obtained by dividing the acid value of the split stock by the saponification value of the fatty acids. The saponification value is usually quite constant for the fatty acids from a given type of feed and is therefore determined only when a change is made in the feed stock.

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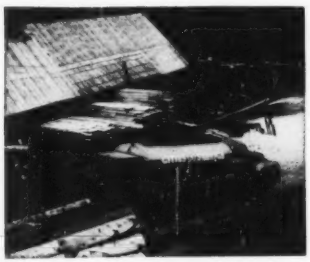

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Splitting is carried out by getting the highest possible FFA consistent with a reasonably high feed rate and glycerol content in the sweetwater. Specific gravity provides a rapid method of determining the glycerol content of the sweetwater for all practical purposes. Where greater accuracy is required, glycerol content is determined by methods employing periodic acid, sodium periodate or potassium dichromate.

Glycerine Recovery

Glycerine is recovered from the splitting operation in the form of sweetwater containing 10% to 15% glycerol. This dilute solution is concentrated to an 88% saponification crude glycerine, which is usually sold as such. Here again specific gravity is usually employed to control evaporator operations. Glycerol content of the finished crude is determined by either the acetin or sodium periodate methods which are alternate official A.O.C.S. methods for the analysis of saponification crude glycerine.

Fatty Acid Distillation

Fatty acids are distilled to improve their color and to remove neutral fat, color bodies, salts and other impurities. The principal control tests at this point are distillate color and residue FFA. Distillate color serves as a control on still performance while residue FFA is an effective control on the residue yield. Many other tests may be made on the distillate, especially if it is being sold as distilled fatty acids rather than being separated into stearic and oleic acid. These tests include titer, iodine value, free fatty acid, unsaponifiable and color stability.

Fatty Acid Separation

Mixed fatty acids obtained by the hydrolysis of fats and oils are commercially separated by fractional distillation, pressing or solvent crystallization. Pressing and solvent crystallization are best adapted to the separation of unsaturated acids, such as oleic and linoleic, from saturated acids, such as palmitic and stearic. Control tests include titer of the liquid acids and iodine value of the solid acids as these tests best indicate the efficiency of separation. Fractional distillation is most effective for separating fatty acids of different chain length, such as lauric, myristic, palmitic and stearic. Acid value and titer are the principal control tests made on the products from this type of separation.

Fatty Acid Finishing

The separated fatty acids are often bleached with activated clay to improve their color. Color is determined spectophotometrically as optical density or percentage transmission at specified wavelengths, or in a Wesson Colorimeter using standard red and yellow Lovibond glasses.

Fatty Acid Products

End products may be blended to meet standard specifications or specific customer requirements. Each lot of finished material is given an analysis number when it is tested for conformance to specifications. Tests made on finished products usually include titer, iodine value, free fatty acid, unsaponifiable, saponification value and color. Resistance to oxidation, color stability and composition tests are made as required. Materials going into lubricating grease may also be tested for oxidation stability by the Norma-Hoffmann test procedure, and for polyunsaturated acid content.

In the foregoing discussion an attempt has been made to acquaint you with the types of tests used by the fatty acid industry for evaluating its raw materials, for controlling the quality of fatty acids during production, and for maintaining the quality of finished products. While many of these tests are found in the Official and Tentative Methods of the American Oil Chemists' Society, a number of important testing procedures developed by the fatty acid industry are not generally known outside the industry. These include (1) rapid methods for determining the glycerol content of fats, (2) the composition of commercial stearic and palmitic acids, (3) a method for checking the color stability of fatty acids at elevated temperatures, (4) measurement of the relative stability of fatty acids against oxidation, and (5) a method for determining the hydroxyl value of such products as hydrogenated castor oil and 12-hydroxystearic acid. The hydroxyl value is applicable to all types of esters but is especially recommended for esters containing a relatively high free fatty acid content. The principal analytical methods for making these tests and the significance of the tests to the fatty acid producer and consumer will now be discussed.

1. Titer

Titer is closely related to solidifying point and may be defined as the temperature at which a fatty acid solidifies under controlled conditions. It is important that the titer test be made on dry fatty acids containing no neutral fat whatsoever. When we say that a fat or oil has this or that titer, we really mean the titer of the fatty acids separated from that fat or oil. Laboratory separation of the fatty acids from a fat is accomplished by saponification of the fat with potassium hydroxide dissolved in glycerine followed by acidulation.

From the titer of an animal fat the fatty acid producer can tell what yields of stearic acid and oleic acid to expect and whether the fat is predominantly beef, pork or a blend of the two. When applied to finished products, particularly the saturated acids, titer is a good indication of the composition of the product. For example, the titers of 100% stearic and palmitic acids are 68.9° C. and 61.7° C. respectively, while the titer of the eutectic mixture is only 53.9° C.

The lubricating grease manufacturer should always include titer in his specifications for distilled fatty acids as it is a good indication of the solid fatty acid content which has a pronounced effect upon grease consistency and should therefore be controlled within reasonably narrow limits.

2. Iodine Value

Iodine value is a measure of the amount of unsaturated fatty acids, such as oleic and linoleic, and when used in conjunction with the polyunsaturated acid content, is useful in establishing the composition of the unsaturated acids.

The Wijs procedure, the official A.O.C.S. iodine value method and the one used throughout the fatty acid industry, measures the absorption of iodine monochloride by the sample, and the results are expressed in terms of the number of centigrams of iodine absorbed per gram of sample. The numerical values obtained are dependent upon the concentration of the reagent, the excess of reagent, the time and temperature of absorption and the presence of light during absorption. Excess reagent is controlled by varying the

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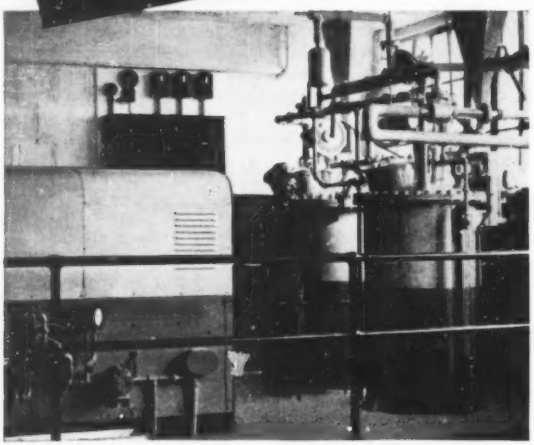

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sample size. For control purposes where it is necessary to get results as soon as possible, as in following the progress of a plant hydrogenation, the absorption time can be substantially reduced by the addition of mercuric acetate catalyst. It should be noted, however, that iodine values obtained with this catalyst on such materials as hydrogenated castor oil and mono-glycerides are in such poor agreement with those run by the official method that catalyst should not be used with such samples.

3. Free Fatty Acid (FFA)

Free fatty acid is a measure of free acid and is calculated as percent oleic acid so as to have a uniform system for expressing the free acidity of all fatty acids. Free fatty acid is probably the most frequently used test in the fatty acid industry. It is of importance as an indication of the quality and approximate glycerine content of raw fats, as a control to measure splitting efficiency, as a control for distillation residue yields and as a measure of purity of finished products.

Acid value also measures free acidity but is expressed as the number of milligrams of potassium hydroxide required to neutralize the acids in one gram of sample.

4. Saponification Value

Saponification value is a measure of the alkali reactive groups in fats, oils and fatty acids and is defined as the number of milligrams of potassium hydroxide required to react completely with all the fatty material present in one gram of a substance. Saponification values are useful for providing information as to quantity and type of glycerides and acids in a given sample. The saponification value provides a direct relative measure of the mean molecular weight of the acids in a sample. Short chain acids such as lauric have high saponification values, whereas longer chain acids such as stearic give correspondingly lower values.

When saponification value is expressed as per cent oleic acid, it is known as total fatty acid (T.F.A.).

For research purposes the saponification equivalent is sometimes used. This test is run in the same manner as saponification value but is calculated as the number of grams of sample which will react with 56.1 grams of KOH. Expressed in this way, the numerical results are a measure of the average equivalent weight of the fatty acids.

5. Unsaponifiable Matter (Unsap.)

"Substances frequently found dissolved in fats and oils which cannot be saponified by the caustic alkalies but which are soluble in ordinary fat solvents" are defined as unsaponifiable matter according to the A.O.C.S. Methods of Analysis. Included are higher aliphatic alcohols, sterols, pigments and hydrocarbons. The unsaponifiable is extracted from an alcoholic solution of the saponified sample with several portions of petroleum ether. The cylinder extraction method is preferable to the alternate method involving the use of separatory funnels.

6. Hydroxyl and Acetyl Values

The hydroxyl and acetyl values are closely related expressions of the number of free hydroxyl groups in a sample. The older acetyl value, which expresses the results as the number of milligrams of KOH equivalent to the acetyl groups in one gram of acetylated sample, has been almost entirely superseded by the hydroxyl value which defines the hydroxyl content on the original sample basis.

The older procedure involved acetylation of the sample, using an excess of acetic anhydride, and measurement of the differences in saponification values of the isolated acetylated product and the original sample. A method more accepted in recent years involves the direct measurement of excess acetic anhydride remaining after reaction of the sample with standard acetic anhydride in pyridine. Variables such as reaction time, excess acetic anhydride, etc., must be carefully controlled. A 2-hour reaction time is used for castor oil and hydrogenated castor oil, while a 4-hour reaction time is used for hydroxylated fatty acids, such as 12-hydroxystearic acid. Five minutes reaction time is usually adequate for ordinary esters and for this reason, the method is particularly useful for following the course of esterification reactions. The method is also applicable to esters containing a substantial amount of free fatty acid.

7. Glycerol Content

Methods for determining glycerol content by the Potassium Dichromate, Acetin and Sodium Periodate Methods are described in the A.O.C.S. Methods of Analysis. Another method, based on oxidation with periodic acid and titration of the excess periodic acid with sodium thiosulfate, is very useful for determining the glycerol content of raw fats and oils. In this method the sample is weighed into an Erlenmeyer flask, saponified with alcoholic potash and then acidulated and heated to remove most of the alcohol. Chloroform is then added and two layers are formed, an upper aqueous layer containing the glycerine and a lower chloroform layer containing the fatty acids. The chloroform layer is then removed and periodic acid added to the flask. After reacting for 10 minutes at room temperature, potassium iodide is added and the sample titrated with sodium thiosulfate using starch as an indicator. A blank is run and the difference in titration between the blank and sample is computed in terms of percent glycerol.

8. Color

Color of raw fat is determined by filling a small glass tube with the melted tallow and comparing it to a series of F.A.C color standards. These standards were developed by the fat analysis committee (F.A.C.) of the A.O.C.S. and are small glass tubes filled with liquids which cover the color range of tallows and greases from a very pale straw to a very dark brown.

For many years it was customary to determine the color of fatty acids using standard red and yellow Lovibond glasses with the Wesson Colorimeter. While this method is still recognized as standard within the fatty acid industry, the many advantages of the spectrophotometric method for determining color are prompting an ever-increasing number of fatty acid producers to employ this method in preference to the Lovibond. Use of the spectrophotometer eliminates the human element which is especially bothersome on color determinations. Moreover, the spectrophotometric method of color measurement is very rapid. A.O.C.S. has a spectrophotometric method for color measurement of soya and cottonseed oils based on determining optical density at four different wavelengths and then substituting the values in an equation to get the photometric color .

Several fatty acid producers have discarded the Lovibond method in favor of a spectrophotometric method whereby

color is expressed as the percent transmission measured at 440 and 550 mmu. 440 mmu gives a good measure of the yellow color while 550 mmu is quite indicative of the red color.

9. Color Stability

Color stability refers to the color of a fatty acid after it has been heated for a time at an elevated temperature. While there is no standard method for performing this test, useful and reproducible results have been obtained by heating a sample in a 1" x 6" test tube held in an aluminum block drilled to hold seven tubes and equipped with thermostatic controls to maintain a temperature of 205 ± 2° C. Unsaturated fatty acids, such as oleic, are heated for one hour and saturated fatty acids, such as stearic, are heated for two hours. Colors are read immediately after the specified heating period.

Color stability tests are useful in predicting how the color of a fatty acid will hold up during such high temperature reactions as esterification.

10. Oxidation Stability

This is a method for testing the susceptibility of fatty acids to atmospheric oxidation. The method involves measurement of the rate of oxygen absorption when a sample of fatty acid is held at an elevated temperature over a period of many hours or days. The number of double bonds present is obviously the most important rate-determining factor but the presence of impurities, such as traces of metal prooxidants, may also greatly affect the velocity of the reaction. The rate of oxygen absorption is generally very slow initially, but when the induction period ends, oxidation becomes greatly accelerated. Under the test conditions, the end of the induction period is arbitrarily taken as the index of relative stability towards oxidation.

11. Composition

Knowing the titer and iodine value of a sample containing an unknown amount of stearic and palmitic acids, Peters and Clark[1] have shown that it is possible to determine the composition of the sample very simply and accurately by blending with a sample of known high stearic or palmitic content. The method is based on use of a graph showing variation of titer for various stearic-palmitic compositions. The method is not applicable to stearic-palmitic blends containing more than 10% oleic acid.

The composition of any mixture of fatty acids can be determined by means of a good fractionating still and an ultraviolet spectrophotometer. One successful type of laboratory fractionating still[2] consists of a column 4 feet in height and approximately 1 inch in diameter packed with a modified Stedman packing, which provides excellent efficiency. Normally, fractionation is carried out at 5 mm absolute pressure and external heaters maintain adiabatic conditions throughout the column. With this unit, it is possible to determine accurately the amounts of each of the saturated acids present in commercial fatty acids. Actually, the methyl esters are fractionated, rather than the fatty acids themselves, since distillation occurs at lower temperatures and decomposition is practically eliminated. The results obtained by interpretation of the distillation curves are usually reproducible within 1.0%. For even greater accuracy, saponification and iodine values may be obtained on each separate fraction and results corrected accordingly.

The polyunsaturated fatty acids, such as linoleic, linolenic and arachidonic, are determined by the A.O.C.S. Ultraviolet Spectrophotometric Method. By comparing the per cent ultraviolet transmissions of unknowns with data for the pure acids, the amount of each polyunsaturated acid can be calculated. The proportions of conjugated and non-conjugated acids can also be computed.

Results obtained from these two analytical devices, together with the usual laboratory determinations of iodine value, saponification value, etc., furnish a very accurate picture of the composition of any commercial fatty acid.

References

[1]Peters and Clark, J. Am. Oil Chem. Soc., 27, 201 (1950).
[2]Weitkamp and Brunstrum, Ibid., 18, 47 (1941).

Effect on Calcium Greases Of Variations in Fatty Acids

George Entwistle
Sinclair Research Laboratories

It has been shown in the preceding papers that the properties of aluminum, lithium and sodium soap greases are materially affected by the type of fatty acids used. Calcium greases differ in this respect from those previously mentioned, in that their properties are not affected by the fatty acids to the same extent.

In manufacturing calcium greases, either vegetable oil fatty acids or animal fatty acids are used. Among the vegetable oils, cottonseed oil is the major source of acids. Calcium greases which are made from straight cottonseed oil fatty acids show a rather poor storage stability, due to their ease of oxidation and poor response to inhibitors. In order to bring about an improvement in this situation, it is a frequent practice to manufacture calcium greases from fatty acids derived from partially hydrogenated cottonseed oil.

Of the animal fatty acids, those derived from tallow are probably used to a greater extent than those which are derived from other sources.

Calcium greases made from animal fatty acids will have dropping points in the region of 220° F., as compared to the 190° F. region which is characteristic of calcium greases made from fatty acids derived from either straight or partially hydrogenated cottonseed oil. Furthermore, the calcium greases of tallow fatty acid derivation are, in general, superior to those of the cottonseed oil fatty acid type in their resistance to oxidation, susceptibility to inhibitors and general storage stability.

A typical specification for the purchase of tallow fatty acids is as follows:

Saponification Number	198-205
Acid Number	196-203
Unsaponifiable Matter	Maximum 1.5%
Iodine Value	55-65
Titer	40-42° C.
NPA Color	Maximum 2½

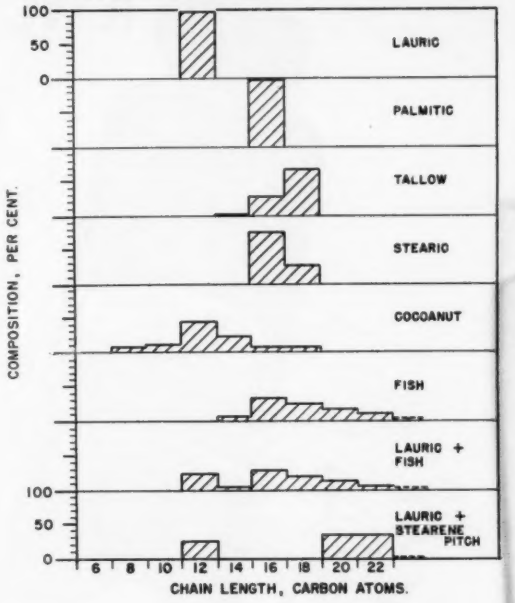
Tallow fatty acids meeting the above specifications are also used as one of the sources of commercial stearic acid and red oil. Typical oidine values of commercial stearic acid and commercial red oil may be assumed to be 6 and 90, respectively. A tallow fatty acid such as specified above would contain approximately from 55 to 65 per cent of red oil, and from 35 to 45 per cent of commercial stearic acid.

When tallow fatty acids showing the extreme limits of iodine value are used to make calcium greases, these greases will be found to have no noticeable differences in structure, stability or yield. However, calcium greases made from commercial stearic acid or red oil will show definite differences in their properties, as compared to each other or to greases made from tallow fatty acids.

To illustrate the foregoing, calcium greases from commercial stearic acid will require about a 20% increase in soap content in order to meet the same penetration as a corresponding grease from tallow fatty acids. Calcium greases made from commercial stearic acid also have a different structure, as evidenced by their crumbly texture and pronounced bleeding. On the other hand, calcium greases from red oil are so much softer that they may be regarded as lacking the gel structure which is normally characteristic of greases.

In conclusion, the essential point originally made may be repeated; namely, that calcium greases are relatively unaffected by moderate variations in the fatty acids normally used in their manufacture. Specifically, the variations in amounts of the commercial stearic acid and red oil occurring in tallow fatty acids are not enough to affect the properties of the grease. The use of commercial stearic acid or red oil, as such, does change the properties of the grease, and the nature of some of these changes has been pointed out.

Figure 1
Composition of Fatty Acids
Suggested for Driving-Journal Grease



Effect of Fatty Acid Variations On the Properties of Soda-Soap Greases

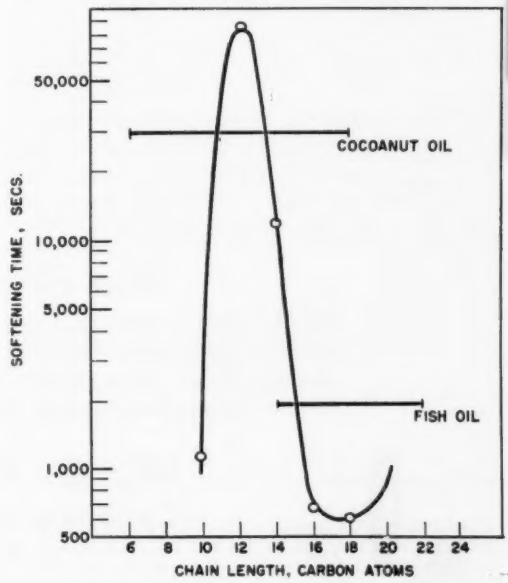
L. C. Brunstrum,
Standard Oil Company (Indiana)

The first part of the N L G I fatty-acid symposium reveals that the complexities of the fatty-acid industry are as numerous as those of the grease industry. If the grease manufacturer desires to avoid a double dose of variables, he must make it his responsibility to describe adequately his requirements to the fatty-acid suppliers. This thesis can be supported by a single example—the effect of fatty-acid composition on the properties of soda-soap driving-journal grease.

Patented compositions illustrate the range of raw materials available for this purpose. Each of these patents was granted upon demonstration of the unique grease properties associated with each fatty-acid type. Selected examples showing the chain-length distribution are given in Figure 1. In this bar chart the location of the bars show what components are present and the height represents the amount.

The critical dependence of grease properties upon fatty-acid composition is shown in Figure 2 for one property—high-temperature consistency, as measured by the softening time test described in U. S. Patent 2,229,042. The curve

Figure 2
Effect of Fatty Acid Chain Length
On High Temperature Consistency



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was developed with individual acids. All greases were made with the same soap content and by the same manufacturing technique. Acids having chain lengths of 12 and 14 carbon atoms yield greases of markedly better high-temperature consistency than those of either 10 or 16 carbons. Other work indicates that for acids of more than 18 carbons, the curve has another upward inflection. As components of commercial mixtures, these acids contribute to the high softening time of greases prepared from cocoanut or fish fatty acids.

Other grease properties studied in the same manner show the results summarized in Figure 3. Because adjacent even-numbered acids yield similar results, the acids may be paired and six-component mixtures may be represented on triangular co-ordinate paper. Thus each point on Figure 3 represents a single fatty-acid composition and the whole chart covers all possible combinations. The solid area represents the composition range of many lots of fatty acids that produced a given brand of grease with satisfactory uniformity. The solid dots, on the other hand, represent individual lots that were offered for the same purpose but that produced grease which was off-test in one respect or another. More divergent compositions, indicated by open circles, were used to correlate composition with grease properties.

The schematic key in the center of Figure 3 represents not a composition but the effect of composition on the more important grease properties. The low-molecular-weight acids produce softer grease, whereas high molecular weight results in harder, somewhat crumbly grease. Both long-chain and short-chain acids promote long softening times. Moving upward on the composition plot improves structure at the expense of high-temperature properties.

Composition of purchased acids may be satisfactorily controlled by specifications, the analyses being accomplished by distillation of the methyl esters. One experience with such a specification may be illustrated by means of the composition-range plot in Figure 4. In this figure the shaded

Figure 3
Relation Between
Fatty Acid Composition and Grease Properties

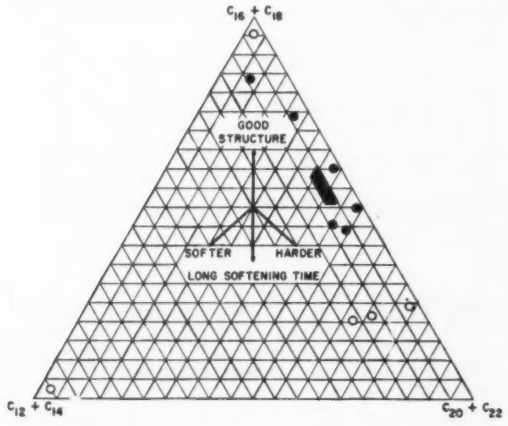
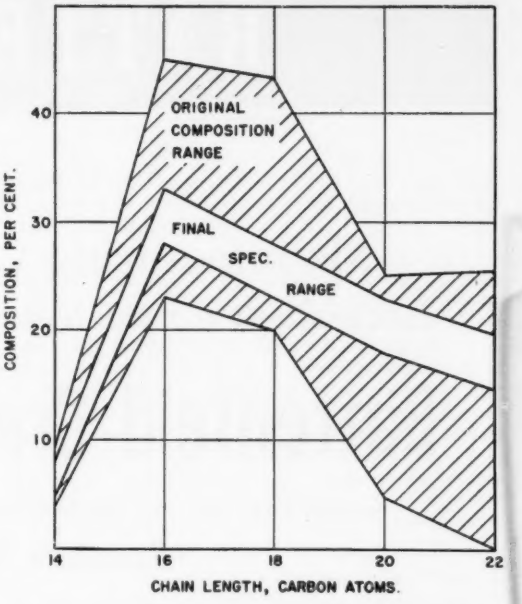


Figure 4
Fatty Acid Specification Based on Composition



area represents the extremes of composition of several lots of four brands of fatty acid. One of the four brands always produced satisfactory grease and always fell in a more restricted range. The other three had similar physical tests but varied in composition and repeatedly produced off-specification grease. Incorporation of the unshaded composition range in purchase specifications resulted in approval of six brands of acid, all of which have consistently produced satisfactory grease.

Fatty-acid composition is thus a fundamental measure of the suitability of the acid for grease manufacturing. Furthermore, it is up to the grease manufacturer to define his needs, because only he has an adequate knowledge of the effect of fatty acid composition on his particular brand of grease. Apparently, the fatty-acid suppliers are able and, within reason, are willing to provide what is required with excellent uniformity.

Some Effects of the Constituent Fatty Acids on the Properties Of Aluminum Greases

Walter K. Dean
Mallinckrodt Chemical Works

The aluminum greases are sensitive to many influences, and it is therefore not surprising to find that their properties are strongly dependent on the nature of the carboxylic acids

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present in the aluminum soaps. Although a considerable amount of work has been done on this subject, particularly by the manufacturers of aluminum stearates, it will be possible to mention only a few of the published or generally accepted relationships in this short paper.

Of the many possible compounds of aluminum with organic acids, the aluminum "stearates" are the most frequently used in grease-making. The "commercial stearic acids" from which they are prepared are, of course, mixtures of a number of aliphatic acids containing twelve to twenty-two carbon atoms, with palmitic and stearic usually predominating. The distribution of chain lengths in these acid mixtures influences the properties of lubricating greases made from their soaps.

Stearic acids derived from animal and vegetable sources do not yield as strongly-gelling aluminum soaps as do those derived from marine fats. A possible explanation is that the fish fatty acids contain a relatively uniform distribution of the higher aliphatic acids and specifically a higher percentage of the C_{20} - C_{22} members. The advisability of using both high- and low-molecular weight fatty acids in grease-forming soaps is commonly known in the art[1]. The beneficial effects of the long-chain organic acids on lubricating porperties and gel stability has been demonstrated by tests on high molecular-weight acids abtained from microcrystalline waxes[2].

Animal and vegetable fatty acids containing higher proportions of palmitic and stearic acids also produce good grease-making soaps, nevertheless. Aluminum soaps made from tallow acids are often characterized by their ability to form smooth, buttery greases under varying conditions of manufacturing.

Although very few commercial aluminum soaps are prepared from pure or nearly pure, single aliphatic acids, the properties of some of these soaps are at least partially known. The aluminum salts of formic, acetic, propionic and butyric acids are not true gel-formers and are therefore of little interest. The capacity to gel seems first to appear in the salts or soaps of the five- and six-carbon acids. Their gelling action is not particularly strong, however. Gelling efficiency then increases with the chain length of the constituent acid, and is considered to reach a maximum when twelve to fourteen carbon atoms are present. It then diminishes somewhat as the chain length is further increased. A possible explanation for the existence of this maximum may be that it represents the best combination of oil-insolubility (due to the presence of the metal) and the hydrocarbon solubility of the organic radical to form a strong colloidal system in the oil. The metal exerts a greater effect when the lower-molecular weight acids are used, limiting the solubility of the compounds; while, if the long organic radicals dominate the soap molecule, it exhibits more nearly the normal solubility characteristics of the fatty acid itself.

It will be understood that the comments above apply mainly to the naturally occurring acids having even-numbered carbon chains. The rare, odd-numbered acids are still too expensive to have received much study in this field. Relatively little is known about the aluminum soaps of branched-chain fatty acids as well, although the di-2-ethyl hexoate seems to form firm gels with good temperature stability and may be superior to the caprylate in these respects.

The introduction of various substituent groups into the fatty acid molecule to increase the structural stability, dropping point and resistance to bleed is a new field that is now receiving quite a bit of attention. Examples of the use of keto and hydroxy acids to impart high dropping points are numerous in the patent literature[4]. The aluminum soap of 12-hydroxy-stearic acid has been reported to increase both the stability and dropping point of aluminum greases[5]. It is probable that such groups can promote the development of a strong polymeric structure in the dispersed soap and thus enhance the strength of the grease.

The aluminum soaps of the dibasic aliphatic acids are not easily dispersed in hydrocarbons, but small amounts of these acids can be used in preparing soaps having special characteristics. For example, aluminum stearates containing minor amounts of dimerized linoleic acid are reported to yield firm, stable greases having a fibrous structure not normally found in products of this type[6]. The stability of these greases may be due to the ability of the dimerized acid to promote cross-linking between the dispersed soap molecules, thus yielding a more durable structure than can be obtained using stearic acid alone. The fibrous structure of the grease suggests that the solubility of the soap is reduced and is beginning to approach that of the alkali and alkaline earth stearates.

The aluminum salts or soaps of the aromatic, alicyclic and unsaturated aliphatic acids are seldom used in the preparation of grease-forming soaps except as minor constituents. The aluminum salts of aromatic acids have been used to increase the hardness and dropping points of aluminum greases, while the naphthenates and oleates, for example, function as smoothing and tackifying agents.

References

[1]Morway, A. J. and Mikeska, L. A., U. S. 2,586,693, Feb. 19, 1952.

[2]Nelson, J. W., U. S. 2,590,786, March 25, 1952 and Teter, J. W. and Nelson, J. W., U. S. 2,590,801, March 25, 1952.

[3]Stingley, N.L.G.I. paper, October 1949.

[4]Morway, A. J. and Smith, D. V., U. S. 2,575,286, Nov. 13, 1951 and Smith, P. V. and Morway, A. J., U. S. 2,589,973, March 18, 1952.

[5]Fraser, H. M., U. S. 2,380,960, Aug. 7, 1945.

[6]American Cyanamid Co., booklet "Aluminum Stearates for Lubricating Greases", 1951.

Lithium Base Lubricating Greases

H. C. Meyer, Jr.
Foote Mineral Company

The lithium soaps of lauric, myristic, palmitic, stearic, arachidic and behenic acids were prepared. From these, greases were compounded by a standardized procedure so that the composition of the soap was the only variable factor.

The greases were then tested to determine whether differences attributable to the fatty acids used could be determined.

The acids used were prepared from commercial fatty acids of known composition. These were subjected to repeated fractional crystallization from ethanol until the calculated molecular weight indicated a minimum of 95% content of the named acid. The maximum iodine value of these acids was 1.0.

The acids at a temperature of 180° F. were then reacted with a 5% aqueous solution of lithium hydroxide. The resultant soap was air-dried at 230° F. for 24 hours at which time the moisture content was less than 0.15%. Free fatty acid content was less than 0.2%; CO_2 less than .01%.

Greases were prepared by dissolving these soaps in oils (Table I) heated to 428° F.

TABLE I

Properties of Oils Used

Type	Vis. S.U.S. 100° F.	VI
Pennsylvania	342	105
California	305	36
Mid Continent	310	70

The dissolving time used was 10 minutes, the proportions 88% oil, 12% soap. 500 grms. of these melts were then cast in 1 liter beakers and allowed to cool and stand 24 hours prior to test. No attempt to influence cooling rate was made.

The greases were worked 60 strokes in the ASTM worker. Penetration and dropping point by the ASTM methods and bleed by the method of ANG3 specification were determined. The greases were then worked a further 500 strokes in the ASTM worker and the penetration redetermined.

TABLE II

PENNSYLVANIA OIL — 12% LITHIUM SOAP

Soap	D.P.-° F.	Bleed-%	Penetration 60 strokes	ASTM 500 strokes
Palmitate	374	8	260	310
Stearate	370	7	240	260
Arachidate	359	5	245	275
Behenate	352	2	220	285

The data presented are significant only for the particular soaps, oils, compositions and processing methods used. There are measurable differences between the members of a given series when the properties are considered in the aggregate. These differences are significant in the terms of this symposium. It will be recognized by experienced grease compounders that such factors as additives, processing methods, mixtures or soaps, mixtures of oils, method of refining and other possible variables encountered in commercial grease manufacture can influence the properties of commercial greases.

TABLE III

CALIFORNIA OIL — 12% LITHIUM SOAP

Soap	D.P.-° F.	Bleed-%	Penetration 60 strokes	ASTM 500 strokes
Laurate	404	8	195—	300
Myristate	395	7	220	255
Palmitate	370	4	225	260
Stearate	368	4	245	275
Arachidate	365	3	255—	300
Behenate	359	2	250	300

TABLE IV

MIDCONTINENT OIL — 12% LITHIUM SOAP

Soap	D.P.-° F.	Bleed-%	Penetration 60 strokes	ASTM 500 strokes
Myristate	383	9	220	305
Palmitate	374	7	220	260
Stearate	368	5	230	260
Arachidate	365	4	250	275
Behenate	359	4	260	300

NOTE: Where no grease was formed soap is not listed.

It would seem desirable, therefore, to reduce the sources of potential variation wherever possible. To indicate the possible variation in acid, consider this specification which is typical of many commercially available fatty acids.

Acid No.	—	199 ± 3
Sap. Value	—	200 ± 3
I.V.	—	6 max.
Titer °C.	—	55° ± 3°

The chemical analysis of a product which would meet these specifications might vary within the following limit:

Myristic Acid	.5% — 8.5%
Palmitic Acid	20% — 8.5%
Stearic Acid	24% — 76 %
Arachidate Acid	0% — 24 %
Behenate Acid	0% — 15 %

It is recognized that all possible combinations within these limits will not meet the specification given. However, an examination of the specifications of commercially available fatty acids will show many products are available meeting this specification.

It may be concluded, therefore, that the chemical composition of the fatty acids used in their preparation can affect the properties of lithium base greases. The physical property type specification currently used for fatty acids does not define their chemical composition. Specifications defining the chemical composition of commercial fatty acids would, therefore, be desirable.

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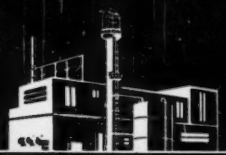

Patents and Developments

Fiber-Length Controlled Grease

The use of gelling agents having anisometric (uneven) fibers produced by controlled processing operations is claimed to give a number of advantages, such as storage stability, increased resistance to shear and bleeding, good thermal reversibility, etc. U. S. Patent 2,614,079, issued to Shell Development Company, describes such a gelling agent consisting of a mixture of fine and coarse fibers in the proportion of 3:1 to 1:3. By fine fibers is meant to include fibers of roughly 0.1 or less microns wide and 4 or more microns in length, while coarse fibers include fibers of 0.5 or more microns wide and over 10 microns in length. By gelling fibers it is meant to include soaps capable of forming fibers such as the alkali and alkaline earth metal soaps, and organic gelling agents such as nylon fibers, cellulose materials, etc.

It has been observed that grease structure is dependent upon the gelling matrix which holds the oil or other suitable base in the composition, which in turn depends upon the fibers lying across one another. Apparently, oil is retained in grease by the capillary attraction of the gelling agent, and bleeding occurs when the liquid head due to gravity exceeds the ability of the capillarity to hold the oil. Gelling agents consisting of oily coarse fibers are claimed to have considerable mechanical stability, but due to the relatively large spaces between fibers, oil tends to flow from the mass. Fine-fibered gelling agents, on the other hand, are claimed to hold oil very well, but often have poor resistance to breakdown on working and, in extreme cases, actually settle out due to lack of stiffness of the fine fibers, and little, if any, consistency is said to be imparted to greases in the sense that such greases are capable of maintaining a given shape against gravity for any length of time. Use of anisometric fibers is claimed to form a cross-linked matrix which not only has high mechanical stability, but also is extremely resistant to bleeding or oil separation.

These greases are claimed to be made in a variety of ways. For example, pre-made soap of fine fiber and pre-made soap of coarse fiber may be admixed in the required proportions. The fiber mixture may be derived from different soaps, e.g. a coarse-fibered sodium soap with a fine-fibered lithium soap.

A particularly suitable method, when the entire gelling agent is to be made of the same soap stock, involves admixing with a portion of the base oil used to make a grease, the saponifiable materials and saponifying agents, and heating the soap in the oil solution formed until the soap has been dehydrated. The soap-oil mixture then is heated to above the melting point of the soap or soap mixtures until a homogeneous mass has been obtained. To form the desired coarse fibers, this concentrate can be cooled slowly to below 100° C. and the coarse fibers thus formed admixed with fine-fibered soap prepared in a similar manner but, instead of slowly cooling the hot homogeneous soap-oil solution, quenching said hot solution to a desired temperature. Sudden cooling to a desired quench point is claimed to form small, fine crystals or fibers.

A combination of the above process consists in forming a grease concentrate containing the soap necessary for the final grease, by heating the saponifiable and saponifying agents in about 30% oil above the melting point of the soap until a homogeneous mass has been obtained (Point A in Figure 1), and then quenching to a desired temperature (Point B), by adding, at a controlled rate, the remaining oil, so that the soap can form fine fibers. The coarse fibers then can be formed by slowly cooling along BD or reheating the grease containing already fine-fibered soap along BC and thereafter cooling slowly along CD to form the final product.

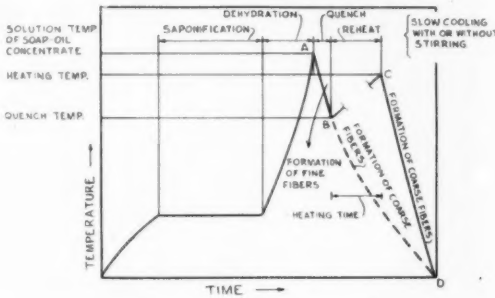

FIG. I

Among the stabilizers which may be used are polyethylene glycol, phenyl alpha naphthylamine, 2, 4-dibutyl 6-methyl phenol, lithium gamma hydroxyvalerate, and lithium alpha hydroxypropionate.

Magnesium Oxide-Carbonate Thickened Greases

Although a number of inorganic thickeners for greases have been proposed, many of them possess important disadvantages. For instance, carbon black-thickened greases are extremely black and have the tendency of staining hands and clotting excessively. Greases thickened with bentonite and silica gels are claimed to be unduly abrasive. In U. S. Patent 2,614,986, Standard Oil Development Company discloses the use of magnesium oxide and carbonate as particularly suitable for the preparation of light colored greases. Proportions of 10-25% by weight are required, whereas in the case of the zinc or calcium compounds, as much as 50-60% would be needed to produce the same thickening effect.

A grease given as an example, containing 18% Baker's light magnesium oxide, had a melting point considerably above 500° F. and showed less wear on the Shell 4-Ball Machine than several compounded mineral oils containing a polybutene thickener.

Replacing Liquid Component in Greases

According to U. S. Patent 2,616,850 issued to Standard Oil Company (Indiana), it is "frequently" desired to replace the oil in a grease system with another oil which is more viscous or less viscous than that already present, or with a liquid polar component such as glycerine. This is claimed to be accomplished by solvent extraction in liquid phase, preferably at 40°-100° F., followed by addition of the replacement vehicle and evaporation of residual solvent.

In one example, a lithium soap grease containing 7.3% soap and an oil having an S. S. U viscosity of 100° F. of 360 seconds, was extracted with hexane until practically all of the petroleum oil was removed. A silicone polymer oil of 300 seconds viscosity at 100° F. in equivalent volume was then added to the soap-hexane system and the solvent evaporated.

The recovered silicone grease had a consistency similar to that of the original grease and had a micropenetration of 98 compared to 110 for the original grease.

Alkaline Earth Metal Complexes of Increased Metal Content

A series of somewhat voluminous patents (2.616,904-6 and 2,616,911, and 2,616,924-5) were recently issued to The Lubrizol Corporation on novel organic metal complexes which are claimed to be useful in detergent oils, greases, and other lubricants.

According to these patents, the greatest total amount of alkaline earth metal which can possibly be incorporated in so-called "basic soaps" by prior art methods has been obtained when using barium and, in that case, it is equal to about 2.3 times the theoretical amount present in the normal salt. The method proposed in the patents is claimed to give a higher "metal ratio" (the ratio of the total metal in the complex to the amount of metal which is in the form of the normal salt of the oil soluble organic acid).

In this process, the organic acid compound (i.e. the acid or salt) is caused to combine with a salt-forming metal compound (under conditions which would produce a normal salt) by increasing the amount of metal that would be thus combined, by having present in the process mass:

(a) An amount of such metal compound substantially in excess of the stoichiometric amount required to form the normal metal salt;

(b) An organic compound selected from the class consisting of those organic compounds which are water soluble at 50° C. to the extent of at least 0.0005% and which, in the presence of water, have an ionization constant greater than about 1×10^{-10} at about 25° C.;

(c) Water (free or combined); and maintaining the mass at a temperature and for a period of time sufficient to drive off all free and hydrated water.

The following example is illustrative of the process disclosed.

In a flask are placed 1980 gms. of the barium salt of petrolatum sulfonic acid (9.2% sulfate ash, and derived from Pennsylvania amber petrolatum), and 132 gms. of p-tert. butyl phenol, and they are heated to 95° C. To this mixture are added 417 gms. of barium oxide and 100 gms. of water, as a slurry, and the mixture is refluxed for 1 hr. Then the temperature is raised slowly to 150°-160° C. and maintained there for 1 hr. to remove all water. The product is filtered to yield a salt complex which is a light brown viscous liquid, clearly soluble in Pennsylvania 150 neutral oil, with Basic No. 23.1 Metal ratio 1.70 and 13.96% Sulfate Ash.

The preferred acid under (b) above, is diisopropyl salicylic acid.

In U. S. Patent 2,616,096, component (b) is an enolic compound such as an aliphatic polynitro compound, oxime, imine, imide, anide, ketoester, polyester or polyketone, and, it is called a "promoter". In Patent 2,616,924, component (b) preferably is an alkyl phenol, while in Patent 2,616,925 it is an alkaline earth metal salt of di-n-butyl dithio phosphoric acid.

News Items

Raybo Chemical Co., Cleveland, introduced a new product (77-Algel) which is claimed to eliminate the need for heat in the production of aluminum octoate gel structures in many petroleum products (Oil, Paint & Drug Rep. September 22, 1952 page 75).

Reynolds Metals Co. is offering a white, organic-free premium grade aluminum trihydrate for the petroleum and other industries (N. Y. J. Comm. December 15, 1952, page 10).

U. S. Patent 2,620,114 (Graham)—Filling device for grease guns.

Technical Committee

Chairman T. G. Roehner,
Director of the Technical Service Department,
Socony-Vacuum Laboratories

It will be recalled that a questionnaire was sent to all members of the N L G I Technical Committee requesting their suggestions regarding a subject for the 1953 symposium to be held in October during the N L G I Annual Meeting. The replies to that questionnaire have been summarized. The subject which received the most votes was "Dispensing of Lubricating Greases in Service Stations and Garages". The next most popular subject was "Recent Developments—Such as Non-Soap Type Greases and Additives". "Manufacturing Methods" and "Laboratory Methods" also received considerable support. Some suggestions stated in effect that the subject of the 1952 symposium, namely "Utilization of Fatty Acids in the Manufacture of Lubricating Greases", should be continued for the forthcoming symposium.

In light of the outcome of the tabulation of the replies to the questionnaire, it has been decided that the subject for the symposium in October shall be "Dispensing of Lubricating Greases in Service Stations and Garages". A committee is being organized to handle the details. In the meantime, H. L. Hemmingway, L. W. McLennan and T. G. Roehner would welcome any suggestions regarding speakers to discuss phases of the main subject.

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Andrew MacLeish Distinguished Service Professor Emeritus, University of Chicago

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1952, 425 pages, $10.00

Performance of Lubricating Oils

by H. H. ZUIDEMA
Shell Oil Company Research Laboratory, Wood River, Illinois

Amply supplied with helpful charts, tables and illustrations, this new book summarizes and evaluates the wealth of data on the performance characteristics of lubricating oils under various conditions. Here, you'll find discussions not only of general lubrication requirements, but **entire chapters** devoted to the lubrication process, flow characteristics, sludge and lacquer deposition, emulsification and foaming, and other important topics. In addition, a special chapter points out the effects of each step in the manufacturing process upon the performance characteristics of the final product.

American Chemical Society Monograph, 1952, 250 pages, $5.00

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by A. BONDI
Shell Development Company, Emeryville, California

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1951, 450 pages, $10.00

Motor Oils and Engine Lubrication

by CARL W. GEORGI
Technical Director, Research Laboratories, Quaker State Oil Refining Corp.

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PEOPLE in the Industry

Martin Goland Chosen To Work With Office Of Ordnance Research

Martin Goland, associate director for engineering at Midwest Research Institute, has been elected a Regional Councilor of the Office of Ordnance Research, it was announced. He will also serve as a member of the Engineering Advisory Committee of this vital defense organization.

Mr. Goland, as one of eleven regional councilors chosen from leading scientists in the United States, will act as liaison between the Ordnance Corps and universities and other institutions throughout the Middle West. In this capacity he will evaluate and correlate the requirements, scope and objectives of the government's ordnance research program. Working closely with the educational institutions in the central states, he will coordinate basic research investigations to be conducted by many of these organizations.

According to Colonel W. W. Holler, commanding officer of the Office of Ordnance Research, Mr. Goland's appointment to this post will be of great assistance in "providing a bridge between pure science and the practicing engineer".

The Office of Ordnance Research is the advanced research group of the U. S. Army Ordnance Corps in the fields of armor, weapons, ammunition, tactical, supply and special-purpose vehicles, pyrotechnics, fuels and lubricants, soil trafficability, human engineering, and associated problems.

Bruce K. Brown Will Speak To Group in Wichita

Bruce K. Brown, former deputy administrator of the Petroleum Administration for Defense and president of the Pan-Am Southern Corporation of New Orleans, will be the principal speaker at a dinner in Wichita, Kansas, on Friday, March 20, when the Vickers Petroleum Company of that city dedicates its multi-million dollar refinery additions, featuring a new thermofor catalytic cracking unit.

The ceremonies will begin at the Lassen Hotel in Wichita, where the 150 invited guests will be picked up in cars for a trip to the Vickers refinery at Potwin, Kansas, in the afternoon.

At the refinery, the Southwestern Engineering Company of Los Angeles will be given a plaque in recognition of its completion of the new thermofor unit in less time and at a lower cost than estimated.

After an inspection of the new facilities, the party will return to the Lassen Hotel for a cocktail party to be followed by a dinner.

Other speakers at the dinner will be Jack A. Vickers, Jr., Robert P. Miller and a top executive representing the Socony-Vacuum Company of New York, licensors of the new airlift T.C.C. unit.

Lincoln Announces Personnel Changes

A. J. GRUENEWALD T. V. PICRAUX R. E. REDENBAUGH

Lincoln Engineering Company, St. Louis, announces the promotion of T. V. Picraux to the position of production manager of both the company's plants, assuming full charge of the production department's operating staff and plant facilities.

Mr. Picraux was formerly sales manager of Lincoln's industrial division, and previous to his five years in that position, was a production executive.

Albert J. Gruenewald, formerly a sales engineer for the company, replaces Mr. Picraux as sales manager of the industrial division.

Robert E. Redenbaugh has been appointed manager of the original equipment division. He will direct sales of the company's lubricant application devices to the original equipment market. Mr. Redenbaugh joined the company in 1942, and served in the product design and sales engineering departments until his present appointment.

API Appointments Made On Jobber Subcommittee

The appointment of James E. Dyer, vice-president of Sinclair Oil Corp., New York, as chairman of the Jobber Advisory Subcommittee of the Division of Marketing of the American Petroleum Institute was announced by R. M. Bartlett.

Mr. Bartlett, general sales vice-president of Gulf Oil Corp., Pittsburgh, is the API vice-president for the Division of Marketing. He served as chairman of the jobber subcommittee last year.

Mr. Bartlett also announced the appointment of R. C. Arnold, general sales manager, Gulf Oil Corp., Pittsburgh, as a member of the jobber subcommittee. He said all other members of the committee were re-appointed for 1953.

Director of Sales Named by Witco Chemical Co.

MAX A. MINNIG

Max A. Minnig, vice-president and national sales manager of rubber chemicals for Witco Chemical Company, has been made director of sales for the company. Mr. Minnig joined the company in 1946 in the Natural Gas Division, Carbon Black Operations.

Appointed assistant to Mr. Minnig is William Wishnick who came to Witco in 1949 after graduating from the University of Texas. He began his career with Witco in the Chicago asphalt sales division.

Maisch Elected Treasurer Of E. F. Houghton & Co.

John F. Maisch was elected treasurer of E. F. Houghton & Co., Philadelphia, by the board of directors at the organization meeting following the annual stockholders meeting in Philadelphia in February.

Mr. Maisch has been controller of the company since June, 1950, when he came to Houghton from Lybrand, Ross Bros. and Montgomery, a Philadelphia accounting firm. He succeeds Wm. F. MacDonald who has been holding the offices of president and treasurer but has relinquished the latter in order to give more time to his duties as president.

At the annual stockholders' meeting Major A. E. Carpenter was reelected chairman of the board, and Wm. F. MacDonald was renamed president. Directors also reelected are: Mrs. E. A. Carpenter, A. E. Carpenter, Wm. F. MacDonald, R. H. Patch, D. J. Richards, C. H. Butler, H. B. Fox, J. T. Eaton, D. C. Miner, and W. K. Barclay.

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Appointments Made By Alemite Division

Three appointments to newly created positions in the factory sales organization of the Alemite division of Stewart-Warner Corporation have been announced by Gus Treffeisen, distribution sales manager.

Huntington Eldridge, who has headed automotive and farm market sales, has been made assistant distribution sales manager. He will continue to carry his previous automotive and farm market sales responsibilities.

H. J. Howerth, formerly in the wholesale or original equipment sales division as head of the Detroit branch, has been made head of farm market sales development. In this newly created post he will develop programs for the marketing of lubrication products by Alemite distributors through farm implement dealers and other farm outlets.

G. W. Mullin, for the past two years in the Los Angeles Alemite distributorship, has joined the factory sales organization as a national accounts representative. A veteran of twenty years in the automotive lubrication equipment field, Mr. Mullin will handle sales to national accounts in a newly set-up territory comprising several Southwestern states.

John D. Farr Joins Emery Industries Staff

JOHN D. FARR

Emery Industries, Inc., announces the appointment of John D. Farr to its development and service department.

Mr. Farr, a chemical engineering graduate of the University of Illinois, has had considerable experience in vinyl plastics and synthetic rubbers with the B. F. Goodrich Company, at both its Akron and Marietta, Ohio, plants.

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- Its planning indecisive.

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Industry NEWS

Alemite Division Announces Automatic Lubricator For Conveyor Trolley Wheels

Lubrication of conveyor trolley wheels, long either neglected or done by hand with resultant conveyor stoppage, waste of manpower and lubricant, and risk of injury to workmen performing the job, has been made automatic, announces the Alemite division of Stewart-Warner Corporation.

The new Alemite lubricator delivers a pre-set, constant shot of either oil or lubricant to each trolley wheel on each side of the conveyor system into which it is installed by the flick of a switch. When every wheel in the system has been serviced, the system may be switched off and is in readiness to go into operation again at whatever lubrication interval load and other conditions require.

The entire unit is contained on a section of I-beam 37¾ or 40 inches in length, which is fitted into the conveyor track at any convenient spot in its length, so long as the location is within reach of the house air line and a 115-V., 60-cycle electrical outlet. Head-room of 22 inches over the top of the I-beam is required.

As each wheel reaches the spot on the track at which the lubricator is installed, a coupler clamps the lubricant fitting on its hub and delivers the required amount of lubricant or oil. The coupler instantly disengages and is ready for the next wheel.

As the applicator and coupler engage the grease fitting on each wheel, a pressure of from 800 to 1000 pounds is applied which forces the lubricant into the bearing. The amount of lubricant can be adjusted to .005 cu. in.—enough for 32,000 shots from the five pound grease or 2½-quart reservoir.

Wheel spacing is no problem—they can be yards apart, irregular in spacing or as close as 8" on centers, and it is as easily adaptable to wheels three inches in diameter as four or six inches.

When grease is used, the house air is regulated to apply pressure to a follower plate on the lubricant to assure delivery of lubricant to piston plunger.

The new lubricator, sold as a "package unit" complete with a section of track, also includes the lubricant reservoir, right and left hand lubricator assemblies with protective covers and anchor plates. As an accessory to the trolley wheel lubricator, Alemite's oil mist system can be installed to automatically lubricate chain linkage and knuckle joints of load pendant hooks.

LUBRICATE FOR SAFETY— EVERY 1000 MILES

Emery Offers Booklet on 12-Hydroxystearic Acid

A booklet describing Emery's 12-Hydroxystearic Acid and Hydrogenated Castor Oil is now available upon request.

The descriptive booklet contains their physical and chemical properties, specifications, and suggested uses. A complete bibliography is also included.

For a copy of this booklet, write to:

Emery Industries, Inc.
Dept. 5, Carew Tower
Cincinnati 2, Ohio

W. C. Hardesty Produces Hydrogenated Castor Oil In Its Canadian Plant

Hydrogenated castor oil is now available from Canadian production according to a recent announcement by W. C. Hardesty Co. of Canada Limited. W. C. Hardesty claims to be the first in Canada to produce this product. The product is a hard waxy solid with the unusually high melting point of 180° F. It is soluble in most organic solvents but highly insoluble in water.

Its main component is the glyceride of 12 hydroxy stearic acid. The hydroxyl group of the component hydroxy stearic acid allows this product to undergo some of the reactions of a fatty alcohol in addition to all the normal reactions of a triglyceride.

A few of the product's important uses are in all purpose greases, paper (waterproof coatings), polishes, electrical insulations, protective coatings, inks, cosmetics, pharmaceuticals, candles and leather dressings.

A complete bulletin on this product is available from W. C. Hardesty Co. of Canada Limited upon request.

Theme and Slogan Chosen For 'Oil Progress Week'

Conservation will be the theme, and "Oil—at Your Service" will be the slogan for this year's observance of Oil Progress Week.

This was announced by the Oil Industry Information Committee, which sponsors the annual event on behalf of the entire petroleum industry.

This year's observance will be held October 11-17.

It will mark the sixth year in a row that the oil industry has opened its doors wide, and invited the public to inspect its operations and policies, facilities and progress. Thousands of oil men from coast to coast participate in the observance, and stage many special events to attract public interest.

Stanton K. Smith, president of Smith Oil & Refining Co., Rockford, Ill., and 1953 national chairman of the Oil In-

dustry Information Committee, commented:

"The oil industry long has been an exponent of conservation of our natural resources.

"Through the years, it has learned how to recover, transport and process petroleum with a minimum of waste and a maximum of efficiency.

"Research and competition have provided the American people with a ceaseless flow of new and improved products. Likewise many other benefits in time, equipment and services have resulted from conservation practices.

"True conservation provides for not only preservation of our natural resources, but also full utilization of those that are withdrawn for consumption.

"With this as the theme, we hope to give the people a far-reaching look at the oil industry, and the bright future it has inspired through true conservation of one of our chief natural resources."

Mr. Smith also announced that the OIIC has set up a special subcommittee to further expand participation by oil men and companies in the year-around program of the national committee. He explained:

"More than 20,000 oil men are taking part in our activities now. They all are volunteers and we are proud of them and their efforts in helping to keep the public informed about the oil industry and its progressiveness. But we can always use more. Obviously, the more who participate, the more effective will be our whole program."

Membership of the Participation Subcommittee follows:

Ralph Carey, Shell Oil Co., Boston, chairman; W. J. Loufman, Fleet Wing Corp., Cleveland, O.; W. Chalmers Burns, Hartol Petroleum Corp., New York; Charles Z. Hardwick, The Ohio Oil Company, Findlay, O.; Frank V. Martinek, Standard Oil Company (Indiana), Chicago, and Charles A. Perlitz, Jr., Continental Oil Co., Houston, Tex.

This subcommittee has been asked to make a study of present participation by individuals and companies, and make recommendations as to what steps are necessary to expand their activities.

Continental Can Acquires Assets and Business Of B. C. Betner Co.

Continental Can Company has acquired the assets and business of the Benjamin C. Betner Company of Devon, Pa., in exchange for 73,177 shares of common stock, it has been announced by General Lucius D. Clay, chairman of the board of Continental Can Company.

The Betner company manufactures small and medium-size bags from various materials including paper, foils, cellophane, laminates, glassine, plastics and wax paper. In addition to the plant at Devon, the company operates plants at Richmond, Va.; Paris, Texas; Beaumont, Texas, and Los Angeles, Calif.

It also has two wholly-owned operating subsidiaries: Benjamin C. Betner Company of California and Thomas M. Royal & Co. It has 50 per cent stock ownership in the Benjamin C. Betner Company of Wisconsin.

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Unsaponifiable (Wool Grease Alcohols)	6% max.
Saponifiable	94%
Free Fatty Acid (as oleic)	55-60%
Actual Free Fatty Acid Content	90%
Saponification No.	120-130
Free Inorganic Acid	0.2% max.
Iodine Value	20-40
Apparent Solidification Point (titre)	Approx. 44° C.
Softening Point	45-48° C.
% Sulfur	No corrosive sulfur

A.O.C.S. Methods

"Continental will operate this business as a separate division and as a supplement to the company's Fibre Drum and Paper Container divisions," General Clay said.

ASTM Publishes Manual Of Engine Test Methods For Rating Fuels

The second edition of the ASTM Manual of Engine Test Methods for Rating Fuels incorporates all of the changes in the five standard methods for rating motor, aviation, and Diesel engine fuels that have been adopted since their earlier publication in 1948. The six extensively revised supplements contain information on the best practices currently in use for laboratory facilities, installation of the engine test units, provisions for reference materials and electrical, water and air services, and the operation and maintenance of the units.

An important improvement in the manual is the inclusion of an extensive index. Users will find the index of considerable service in locating needed information quickly and conveniently.

Two methods apply to motor fuels: Standard Method of Test for Knock Characteristics of Motor Fuels by the Motor Method (D 357-49) and Standard Method of Test for Knock Characteristics of Motor Fuels by the Research Method (D 908-51). These methods describe the test procedures for determining the knock characteristics of fuels for use in spark-ignition engines, the first in terms of ASTM motor octane numbers, the second in terms of ASTM research octane numbers. An important modification in the "motor" and "research" methods provides for the use of a detonation meter as an alternate for the "bouncing pin" described in the earlier edition to determine knock intensity.

Two methods apply to aviation fuels: Tentative Method of Test for Knock Characteristics of Aviation Fuels by the Aviation Method (D 614-49 T) and Tentative Method of Test for Knock Characteristics of Aviation Fuels by the Supercharge Method (D 909-49 T). These methods describe the test procedure for determining the knock characteristics of fuels for use in aircraft engines of the spark-ignition type, the first at a lean fuel-air ratio, the second at knock limited power under supercharge rich-mixture conditions.

The last method applies to Diesel fuels: Tentative Method of Test for Ignition Quality of Diesel Fuels by the Cetane Method (D 613-48 T). This method describes the test procedure for determining the ignition quality of Diesel fuels in terms of ASTM Cetane number.

All of the methods have come into widespread use and are of great industrial significance, not only to the suppliers of fuels, but also to users of fuels and designers of engines.

Copies of this 360-page Manual, green cloth binding, can be obtained from the headquarters of the American Society for Testing Materials, 1916 Race Street, Philadelphia 3, Pa., at $8.00 each.

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2,568,569
2,568,570

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Standard Ultramarine & Color Co.

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FUTURE MEETINGS of the Industry

MARCH, 1953

15-19 American Chemical Society (123rd national meeting), Hotels Biltmore and Statler, Los Angeles, Calif.

15-19 American Institute of Mining and Metallurgical Engineers (annual joint meeting of petroleum metals, and mining branches), Statler Hotel, Los Angeles, Calif.

16-20 National Assn. of Corrosion Engineers (1953 conference and exhibition), Hotel Sherman, Chicago, Ill.

17-19 Ohio Petroleum Marketers Assn. (annual convention and exposition), Deshler-Wallick, Columbus, Ohio.

18-20 American Petroleum Institute (Division of Production, Mid-Continent District spring meeting), Mayo Hotel, Tulsa, Okla.

19-21 Texas Oil Jobbers Assn. Inc. (annual convention), The Plaza Hotel, San Antonio, Tex.

23-25 Western Petroleum Refiners Assn. (annual meeting), Plaza Hotel, San Antonio, Tex.

23-26 American Assn. of Petroleum Geologists—Socy. of Economics Paleontologists and Mineralogists—Socy. of Exploration Geophysicists (joint annual meeting), Coliseum, Houston, Tex.

24-27 Greater New York Safety Council (23rd annual safety convention and exposition), Hotel Statler, New York, N. Y.

25-27 Socy. of Automotive Engineers (national production meeting), Hotel Statler, Cleveland, Ohio.

28 North Texas Oil & Gas Assn. (annual meeting), Kemp Hotel, Wichita Falls, Tex.

30-31 Texas Independent Producers & Royalty Owners Assn. (7th annual meeting), Gunter Hotel, San Antonio, Tex.

APRIL, 1953

8-10 American Petroleum Institute (Division of Production, Eastern District spring meeting), Hotel William Penn, Pittsburgh, Pa.

13-15 American Socy. of Lubrication Engineers (annual meeting and exhibit), Hotel Statler, Boston, Mass.

13-15 Assn. of Nebraska Liquefied Petroleum Gas Dealers (annual convention), Hotel Fontenelle, Omaha, Neb.

15-17 National Petroleum Association (semi-annual meeting), Hotel Cleveland, Cleveland, Ohio.

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APRIL, 1953

16-19 National Tank Truck Carriers Inc., Boca Raton Hotel, Boca Raton, Fla.

20-22 American Petroleum Institute (Division of Transportation, products pipeline conference), Hotel Muehlebach, Kansas City, Mo.

26-29 American Inst. of Chemical Engineers (joint meeting with Chemical Institute of Canada), Royal York Hotel, Toronto, Canada.

27 API Safety and Fire-Protection Committees, 6th mid-year conference, Galvez Hotel, Galveston, Texas.

27-28 Independent Petroleum Assn. of America (midyear meeting), Jefferson Hotel, St. Louis, Mo.

MAY, 1953

3-5 American Petroleum Institute (Division of Marketing, spring meeting), Baker Hotel, Dallas, Tex.

4-6 Liquefied Petroleum Gas. Assn. (annual convention & trade show), Conrad Hilton Hotel, Chicago, Ill.

7-8 American Petroleum Institute (Division of Production, Rocky Mountain District spring meeting), Gladstone Hotel, Casper, Wyo.

10-12 tentative Pennsylvania Petroleum Assn., Inc. (3-day meeting), Bedford Springs Hotel, Bedford Springs, Pa.

11-13 American Petroleum Institute (Division of Marketing, Lubrication Committee meeting), Greenbrier Hotel, White Sulphur Springs, West Va.

11-14 American Petroleum Institute (Division of Refining, 18th mid-year meeting), Hotel Commodore, New York, N. Y.

11-15 National Fire Protection Assn. (annual meeting), Edgewater Beach Hotel, Chicago, Ill.

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MAY, 1953

14-23 International Petroleum Exposition, Tulsa, Okla.

17-19 Empire State Petroleum Assn., The Roosevelt, New York, N. Y.

28-29 Western Petroleum Refiners Assn. (regional meeting), Broadview Hotel, Wichita, Kans.

JUNE, 1953

2-3 American Petroleum Institute (Division of Production, Pacific Coast District spring meeting), Hotel Statler, Los Angeles, Calif.

4-5 Kentucky Oil & Gas Assn. (annual meeting), Lafayette Hotel, Lexington, Ky.

7-12 Socy. of Automotive Engineers (summer meeting), The Ambassador and Ritz-Carlton, Atlantic City, N. J.

15-19 American Petroleum Institute (Division of Production, midyear committee conference), Hotel William Penn, Pittsburgh, Pa.

18-19 Pennsylvania Grade Crude Oil Assn. (annual meeting), William Penn Hotel, Pittsburgh, Pa.

18-19 Western Petroleum Refiners Assn. (regional meeting), Conrad Hilton Hotel, Chicago, Ill.

28 to July 2 Petroleum Equipment Suppliers Assn. (annual meeting), Broadmoor Hotel, Colorado Springs, Colo.

29 to July 3 American Socy. for Testing Materials (annual meeting), Chalfonte-Haddon Hall, Atlantic City, N. J.

AUGUST, 1953

17-19 Socy. of Automotive Engineers (national West Coast meeting), Georgia Hotel, Vancouver, B. C., Canada.

SEPTEMBER, 1953

6-11 American Chemical Society (124th national meeting), Conrad Hilton Hotel, Chicago, Ill.

13-16 American Inst. of Chemical Engineers, Fairmont and Mark Hopkins Hotels, San Francisco, Calif.

14-17 Socy. of Automotive Engineers (National Tractor Meeting and Production Forum), Schroeder Hotel, Milwaukee, Wis.

16 American Petroleum Institute (Division of Marketing, Lubrication Committee meeting), The Traymore, Atlantic City, N. J.

16-18 National Petroleum Assn. (51st annual meeting), The Traymore, Atlantic City, N. J.

21-23 American Trade Assn. Executives (annual meeting), Chalfonte-Haddon Hall, Atlantic City, N. J.

24-25 Western Petroleum Refiners Assn. (regional meeting), Henning Hotel, Casper, Wyo.

27-29 National Assn. of Oil Equipment Jobbers (annual meeting), Neil House, Columbus, Ohio.

29 to Oct. 3 Socy. of Automotive Engineers (national aeronautic meeting), Statler Hotel, Los Angeles, Calif.

OCTOBER, 1953

15-16 Petroleum Marketers Assn. of Texas (annual meeting), Adolphus Hotel, Dallas, Tex.

19-20 Independent Petroleum Assn. of America (annual meeting), Hotel Texas, Ft. Worth, Tex.

19-23 National Safety Congress, Conrad Hilton, Congress, Morrison, Sheraton, Chicago, Ill.

22-23 Western Petroleum Refiners Assn. (regional meeting), Garrett Hotel, Eldorado, Ark.

26-28 National Lubricating Grease Institute, 21st Annual Meeting, Edgewater Beach Hotel, Chicago, Ill.

28-29 Independent Oil Compounders Association, 6th Annual Meeting, Edgewater Beach Hotel, Chicago, Ill.

29-30 Socy. of Automotive Engineers (international production meeting), Royal York Hotel, Toronto, Ontario, Canada.

NOVEMBER, 1953

2-4 Socy. of Automotive Engineers (national transportation meeting), Conrad Hilton Hotel, Chicago, Ill.

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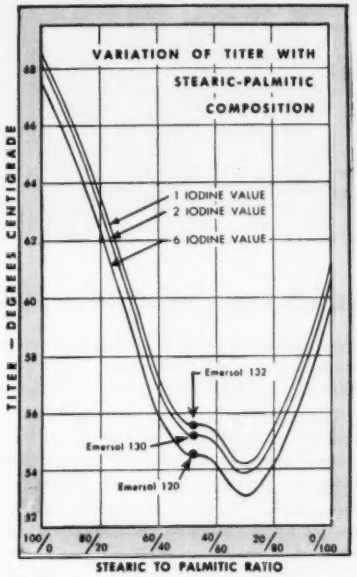

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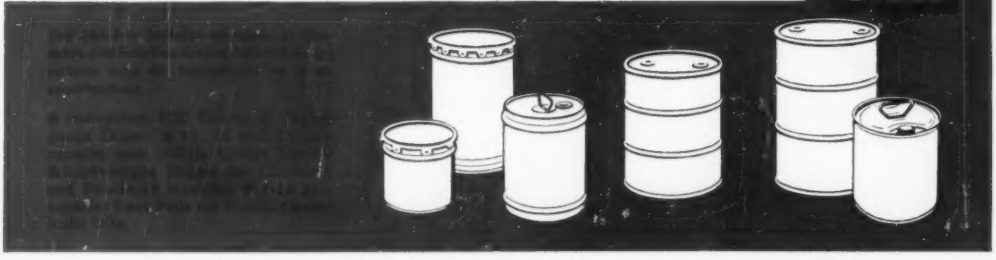

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Representative—Dr. J. J. Whitfield

Witco Chemical Company
75 East Wacker Drive
Chicago 1, Illinois
Representative—E. F. Wagner

CONTAINER AND CLOSURE MANUFACTURERS

Central Can Company
2415 West 19th Street
Chicago 8, Illinois
Representative—Henry Frazin

Continental Can Company, Inc.
1107 Waldheim Building
Kansas City 6, Missouri
Representative—T. A. Graham

Geuder, Paeschke & Frey Company
324 North Fifteenth Street
Milwaukee 1, Wisconsin
Representative—Willard J. Flint

Inland Steel Container Company
6532 South Menard Avenue
Chicago 38, Illinois
Representative—John H. Strome

J. & L. Steel Corp.
(Container Division)
405 Lexington Avenue
New York 17, New York
Representative—Jerry Lyons

National Steel Container Corporation
6700 South LeClaire Avenue
Chicago 38, Illinois
Representative—Henry Rudy

The Ohio Corrugating Co.
917 Roanoke Ave. S. E.
Warren, Ohio
Representative—Lawrence S. McKay

Rheem Manufacturing Company
570 Lexington Avenue
New York 22, New York
Representative—F. J. Blume

Rieke Metal Products Corporation
Auburn, Indiana
Representative—Ralph S. Pearson

United States Steel Products
Division United States Steel Company
30 Rockefeller Plaza
New York City 20, New York
Representative—Wm. I. Hanrahan

Vulcan Stamping & Manufacturing Co.
P. O. Box 367
Bellwood, Illinois
Representative—H. B. Scharbach

MANUFACTURERS OF EQUIPMENT FOR APPLICATION OF LUBRICATING GREASES

Aro Equipment Corporation
Bryan, Ohio
Representative—R. W. Morrison

Balcrank, Inc.
Disney near Marburg
Cincinnati 9, Ohio
Representative—Richard P. Field

Gray Company, Inc.
60 Northeast 11th Avenue
Minneapolis 13, Minnesota
Representative—B. A. Beaver

Lincoln Engineering Company
5701 Natural Bridge Avenue
St. Louis 20, Missouri
Representative—G. A. Hubbard

Stewart-Warner Corporation
Alemite Division
1826 Diversey Parkway
Chicago 14, Illinois
Representative—Walter Duncan

United States Air Compressor Company
5300 Harvard Avenue
Cleveland 5, Ohio
Representative—C. A. Bening

SUPPLIERS OF EQUIPMENT FOR MANUFACTURING LUBRICATING GREASES

Buffalovak Equipment Division of Blaw-Knox Company
1543 Fillmore Avenue
Buffalo 11, New York
Representative—Edward V. Hegg

Chemicolloid Laboratories, Inc.
30 Church Street
New York 7, New York
Representative—David F. O'Keefe

The Girdler Corporation
224 East Broadway
Louisville 1, Kentucky
Representative—J. E. Slaughter, Jr.

Morehouse Industries
707 Henry Grady Building
Atlanta 3, Georgia
Representative—George E. Missbach

Stratford Engineering Corporation
1414 Dierks Building
Kansas City 6, Missouri
Representative—J. W. Sylvester

MARKETING ORGANIZATIONS

California Texas Oil Company, Limited
551 Fifth Avenue
New York 17, New York
Representative—Hal U. Fisher

REFINERS

Farmers Union Central Exchange, Incorporated
P. O. Box G
St. Paul 1, Minnesota
Representative—H. F. Wagner

Freedom Valvoline Oil Company
Box G
Freedom, Pennsylvania
Representative—D. A. Smith

Mid-Continent Petroleum Corporation
Mid-Continent Building
P. O. Box 381
Tulsa, Oklahoma
Representative—J. W. Basore

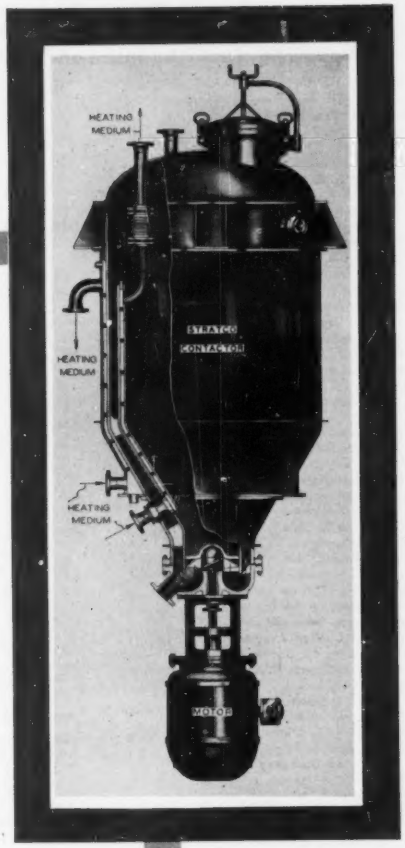
TECHNICAL AND RESEARCH ORGANIZATIONS

Midwest Research Institute
4049 Pennsylvania
Kansas City 2, Missouri
Representative—Dr. M. H. Thornton

Petroleum Educational Institute
9020 Melrose Avenue
Los Angeles 46, California
Representative—G. A. Zamboni

Phoenix Chemical Laboratory, Inc.
3953 W. Shakespeare Avenue
Chicago 47, Illinois
Representative—Mrs. G. A. Krawetz

Sociedade Nacional de Petroleos
Rua D. Pedro V, no. 80
Lisbon, Portugal
Representative—Manuel Corda Boullosa



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